Synthetic, Structural, and Mechanistic Aspects of an Amine Activation Process Mediated at a Zwitterionic Pd(II) Center

Supporting Information

Connie C. Lu and Jonas C. Peters*

Division of Chemistry and Chemical Engineering,
Arnold and Mabel Beckman Laboratories of Chemical Synthesis, California Institute of Technology, Pasadena, CA 91125

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Figure 1. Labeled drawing of 1' (CCDC 171286), with 50% ellipsoids. Hydrogen atoms and solvent molecule (THF) have been omitted for clarity.

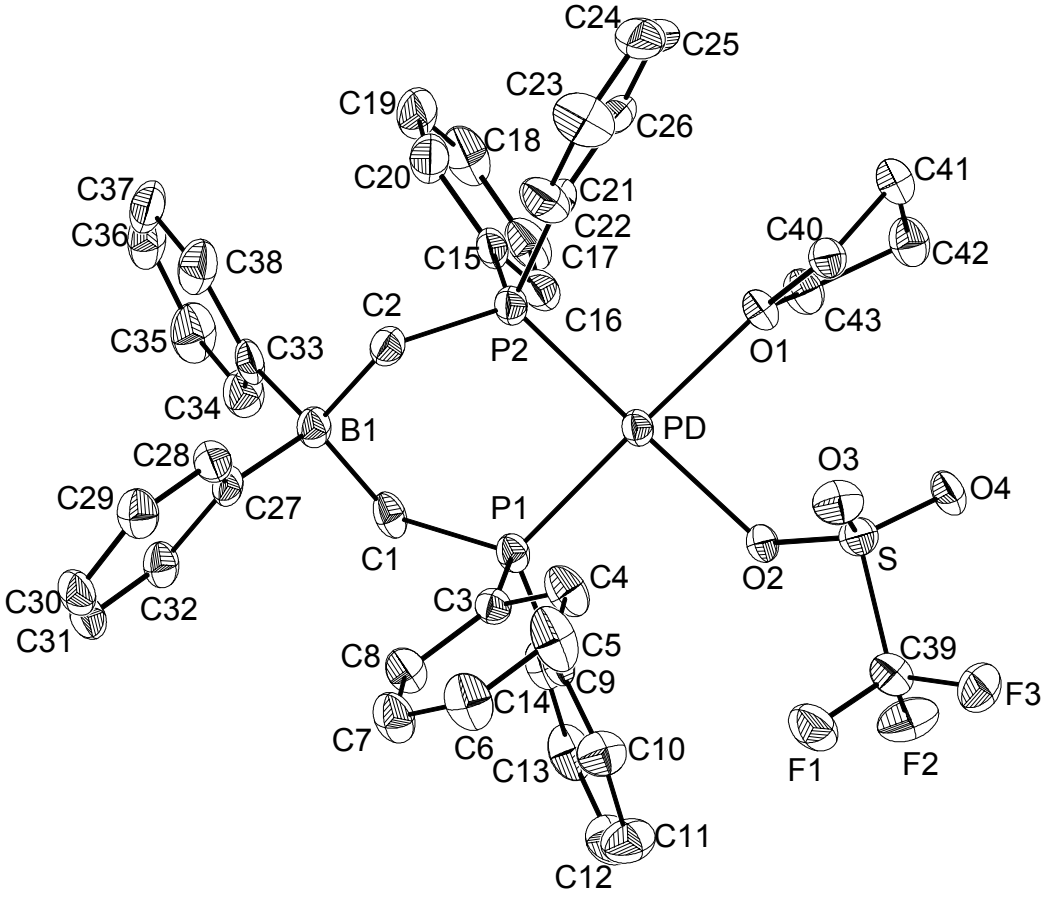

Table 1. Crystal data and structure refinement for 1' (CCDC 171286).

Empirical formula	$C_{43}H_{42}BF_3O_4P_2SPd \cdot C_4H_8O$
Formula weight	963.08
Crystallization Solvent	THF/Petroleum ether
Crystal Habit	Fragment
Crystal size	0.31 x 0.22 x 0.19 mm^3
Crystal color	Colorless

Data Collection

Preliminary Photos	Rotation
Type of diffractometer	CCD area detector
Wavelength	0.71073 Å MoKα
Data Collection Temperature	98(2) K
θ range for 22373 reflections used in lattice determination	2.28 to 27.84°
Unit cell dimensions	a = 13.2666(7) Å b = 17.4832(10) Å β= 95.8550(10)° c = 19.2115(10) Å
Volume	4432.7(4) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/n$
Density (calculated)	1.443 Mg/m^3
F(000)	1984
Data collection program	Bruker SMART
θ range for data collection	1.58 to 28.50°
Completeness to θ = 28.50°	95.0 %
Index ranges	$-17 \le h \le 17$, $-23 \le k \le 23$, $-25 \le l \le 25$
Data collection scan type	ω scans at 7 ϕ settings
Data reduction program	Bruker SAINT v6.2
Reflections collected	90844
Independent reflections	10664 [R_{int}= 0.0815]
Absorption coefficient	0.596 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.8941 and 0.8344

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Patterson method
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	10664 / 10 / 709
Treatment of hydrogen atoms	Unrestrained except on solvent
Goodness-of-fit on F^2	1.743
Final R indices [$I>2\sigma(I)$, 7182 reflections]	R1 = 0.0478, *w*R2 = 0.0701
R indices (all data)	R1 = 0.0805, *w*R2 = 0.0728
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.061
Average shift/error	0.002
Largest diff. peak and hole	1.121 and -0.923 e.Å^{-3}

Special Refinement Details

The crystals contain THF as a solvent of crystallization. The geometry of the solvent THF was restrained to be similar to the geometry of the bond THF and the hydrogen atoms of the solvent THF were restrained to ideal geometry.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 1' (CCDC 171286). U(eq) the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Pd	5290(1)	1940(1)	9470(1)	21(1)
S	3144(1)	1158(1)	9594(1)	27(1)
P(1)	5415(1)	2214(1)	8345(1)	22(1)
P(2)	6602(1)	2713(1)	9768(1)	24(1)
B(1)	7209(3)	3246(2)	8424(2)	25(1)
F(1)	2095(1)	1374(1)	8395(1)	42(1)
F(2)	2393(1)	190(1)	8651(1)	45(1)
F(3)	1279(1)	806(1)	9165(1)	39(1)
O(2)	4043(1)	1170(1)	9202(1)	24(1)
O(3)	2827(2)	1905(1)	9796(1)	33(1)
O(4)	3149(2)	553(1)	10098(1)	30(1)
C(1)	6658(2)	2452(2)	8126(2)	26(1)
C(2)	6792(3)	3477(2)	9175(2)	27(1)
C(3)	4517(2)	2952(2)	8062(2)	22(1)
C(4)	3709(3)	3122(2)	8441(2)	34(1)
C(5)	2988(3)	3645(2)	8190(2)	44(1)
C(6)	3058(3)	4009(2)	7566(2)	39(1)
C(7)	3850(3)	3852(2)	7183(2)	36(1)
C(8)	4578(2)	3324(2)	7428(2)	30(1)
C(9)	5030(2)	1396(2)	7802(2)	24(1)
C(10)	4066(3)	1334(2)	7459(2)	34(1)
C(11)	3818(3)	735(2)	7005(2)	41(1)
C(12)	4535(3)	196(2)	6883(2)	41(1)
C(13)	5487(3)	239(2)	7221(2)	36(1)
C(14)	5743(3)	832(2)	7677(2)	31(1)
C(15)	7741(2)	2149(2)	9949(2)	27(1)
C(16)	7754(3)	1373(2)	9807(2)	30(1)
C(17)	8621(3)	942(2)	9965(2)	37(1)
C(18)	9485(3)	1285(3)	10264(2)	46(1)
C(19)	9492(3)	2057(3)	10406(2)	47(1)
C(20)	8625(3)	2493(2)	10253(2)	37(1)
C(21)	6392(2)	3143(2)	10597(2)	26(1)
C(22)	5860(3)	3824(2)	10607(2)	40(1)
C(23)	5673(3)	4156(3)	11237(2)	48(1)
C(24)	6009(3)	3809(2)	11856(2)	40(1)
C(25)	6534(3)	3132(2)	11858(2)	33(1)
C(26)	6734(2)	2801(2)	11232(2)	29(1)
C(27)	6973(2)	3943(2)	7860(2)	24(1)
C(28)	6580(2)	4649(2)	8022(2)	30(1)
C(29)	6389(2)	5230(2)	7525(2)	33(1)
C(30)	6589(3)	5110(2)	6847(2)	34(1)
C(31)	7001(3)	4420(2)	6669(2)	33(1)
C(32)	7190(2)	3860(2)	7167(2)	28(1)
C(33)	8437(2)	3100(2)	8533(2)	27(1)
C(34)	8915(3)	2476(2)	8263(2)	36(1)
C(35)	9951(3)	2346(3)	8394(2)	44(1)
C(36)	10552(3)	2836(2)	8798(2)	46(1)
C(37)	10124(3)	3473(3)	9055(2)	44(1)

C(38)	9088(3)	3606(2)	8924(2)	39(1)
C(39)	2180(2)	866(2)	8919(2)	30(1)
O(1)	5217(1)	1584(1)	10539(1)	23(1)
C(40)	4637(3)	1847(2)	11105(2)	26(1)
C(41)	4986(3)	1338(2)	11721(2)	30(1)
C(42)	5184(3)	598(2)	11352(2)	32(1)
C(43)	5659(3)	838(2)	10713(2)	29(1)
O(5)	6991(4)	428(2)	5178(3)	173(2)
C(51)	6547(5)	893(6)	5763(3)	200(5)
C(52)	7146(5)	1600(4)	5848(3)	163(4)
C(53)	8060(4)	1384(3)	5518(4)	120(2)
C(54)	7947(5)	706(4)	5101(3)	124(3)

Table 3. Bond lengths [Å] and angles [°] for 1' (CCDC 171286).

Pd-O(2)	2.155 (2)	C(17)-C(18)	1.368(5)
Pd-O(1)	2.157(2)	C(17)-H(17)	0.92(3)
Pd-P(2)	2.2309(9)	C(18)-C(19)	1.377(5)
Pd-P(1)	2.2378(8)	C(18)-H(18)	0.96(3)
S-O(4)	1.434(2)	C(19)-C(20)	1.387(5)
S-O(3)	1.438(2)	C(19)-H(19)	0.88(3)
S-O(2)	1.474(2)	C(20)-H(20)	0.90(3)
S-C(39)	1.800(3)	C(21)-C(22)	1.385(4)
P(1)-C(1)	1.792(3)	C(21)-C(26)	1.392(4)
P(1)-C(3)	1.802(3)	C(22)-C(23)	1.387(5)
P(1)-C(9)	1.812(3)	C(22)-H(22)	0.95(3)
P(2)-C(2)	1.790(3)	C(23)-C(24)	1.368(5)
P(2)-C(15)	1.809(3)	C(23)-H(23)	0.85(3)
P(2)-C(21)	1.809(3)	C(24)-C(25)	1.374(5)
B(1)-C(27)	1.640(4)	C(24)-H(24)	0.96(3)
B(1)-C(33)	1.641(4)	C(25)-C(26)	1.384(4)
B(1)-C(1)	1.645(5)	C(25)-H(25)	0.97(3)
B(1)-C(2)	1.646(5)	C(26)-H(26)	0.92(3)
F(1)-C(39)	1.338(3)	C(27)-C(28)	1.387(4)
F(2)-C(39)	1.330(3)	C(27)-C(32)	1.398(4)
F(3)-C(39)	1.334(3)	C(28)-C(29)	1.399(4)
C(1)-H(1A)	0.93(3)	C(28)-H(28)	1.00(3)
C(1)-H(1B)	0.94(3)	C(29)-C(30)	1.371(4)
C(2)-H(2A)	0.95(3)	C(29)-H(29)	0.96(3)
C(2)-H(2B)	0.90(3)	C(30)-C(31)	1.381(5)
C(3)-C(4)	1.388(4)	C(30)-H(30)	0.91(3)
C(3)-C(8)	1.391(4)	C(31)-C(32)	1.374(4)
C(4)-C(5)	1.375(4)	C(31)-H(31)	0.85(3)
C(4)-H(4)	0.95(3)	C(32)-H(32)	0.90(2)
C(5)-C(6)	1.368(5)	C(33)-C(34)	1.389(5)
C(5)-H(5)	0.85(3)	C(33)-C(38)	1.399(5)
C(6)-C(7)	1.370(5)	C(34)-C(35)	1.391(5)
C(6)-H(6)	0.84(3)	C(34)-H(34)	0.91(3)
C(7)-C(8)	1.384(4)	C(35)-C(36)	1.357(5)
C(7)-H(7)	1.02(3)	C(35)-H(35)	0.91(3)
C(8)-H(8)	0.95(3)	C(36)-C(37)	1.366(5)
C(9)-C(10)	1.383(4)	C(36)-H(36)	0.97(3)
C(9)-C(14)	1.404(4)	C(37)-C(38)	1.391(5)
C(10)-C(11)	1.381(5)	C(37)-H(37)	0.97(3)
C(10)-H(10)	0.87(3)	C(38)-H(38)	0.91(2)
C(11)-C(12)	1.376(5)	O(1)-C(43)	1.454(3)
C(11)-H(11)	0.89(3)	O(1)-C(40)	1.469(3)
C(12)-C(13)	1.361(5)	C(40)-C(41)	1.514(4)
C(12)-H(12)	0.88(3)	C(40)-H(40A)	0.94(3)
C(13)-C(14)	1.378(5)	C(40)-H(40B)	1.02(3)
C(13)-H(13)	0.90(3)	C(41)-C(42)	1.511(4)
C(14)-H(14)	0.96(3)	C(41)-H(41A)	1.03(3)
C(15)-C(16)	1.385(4)	C(41)-H(41B)	1.00(3)
C(15)-C(20)	1.391(4)	C(42)-C(43)	1.496(4)
C(16)-C(17)	1.383(4)	C(42)-H(42A)	0.91(3)
C(16)-H(16)	0.97(2)	C(42)-H(42B)	0.98(3)

C(43)-H(43A)	0.94(3)	B(1)-C(2)-H(2A)	109.5(16)
C(43)-H(43B)	1.06(3)	P(2)-C(2)-H(2A)	103.5(16)
O(5)-C(54)	1.380(5)	B(1)-C(2)-H(2B)	113.3(17)
O(5)-C(51)	1.550(7)	P(2)-C(2)-H(2B)	105.6(17)
C(51)-C(52)	1.470(9)	H(2A)-C(2)-H(2B)	107(2)
C(51)-H(51A)	0.9900	C(4)-C(3)-C(8)	118.5(3)
C(51)-H(51B)	0.9900	C(4)-C(3)-P(1)	121.1(2)
C(52)-C(53)	1.473(7)	C(8)-C(3)-P(1)	120.2(2)
C(52)-H(52A)	0.9900	C(5)-C(4)-C(3)	120.3(3)
C(52)-H(52B)	0.9900	C(5)-C(4)-H(4)	121.5(19)
C(53)-C(54)	1.430(6)	C(3)-C(4)-H(4)	118.2(19)
C(53)-H(53A)	0.9900	C(6)-C(5)-C(4)	120.6(4)
C(53)-H(53B)	0.9900	C(6)-C(5)-H(5)	116(2)
C(54)-H(54A)	0.9900	C(4)-C(5)-H(5)	123(2)
C(54)-H(54B)	0.9900	C(5)-C(6)-C(7)	120.2(4)
		C(5)-C(6)-H(6)	117(2)
O(2)-Pd-O(1)	86.52(7)	C(7)-C(6)-H(6)	122(2)
O(2)-Pd-P(2)	178.42(6)	C(6)-C(7)-C(8)	119.9(3)
O(1)-Pd-P(2)	92.33(6)	C(6)-C(7)-H(7)	120.6(17)
O(2)-Pd-P(1)	91.90(5)	C(8)-C(7)-H(7)	119.5(17)
O(1)-Pd-P(1)	175.29(6)	C(7)-C(8)-C(3)	120.6(3)
P(2)-Pd-P(1)	89.16(3)	C(7)-C(8)-H(8)	119.5(17)
O(4)-S-O(3)	118.04(13)	C(3)-C(8)-H(8)	120.0(17)
O(4)-S-O(2)	114.07(12)	C(10)-C(9)-C(14)	118.0(3)
O(3)-S-O(2)	113.46(12)	C(10)-C(9)-P(1)	121.9(2)
O(4)-S-C(39)	103.42(14)	C(14)-C(9)-P(1)	119.9(3)
O(3)-S-C(39)	104.22(14)	C(11)-C(10)-C(9)	120.7(3)
O(2)-S-C(39)	100.83(13)	C(11)-C(10)-H(10)	120.2(19)
C(1)-P(1)-C(3)	111.06(15)	C(9)-C(10)-H(10)	119.1(19)
C(1)-P(1)-C(9)	105.07(15)	C(12)-C(11)-C(10)	120.1(4)
C(3)-P(1)-C(9)	104.65(14)	C(12)-C(11)-H(11)	120.7(19)
C(1)-P(1)-Pd	115.89(12)	C(10)-C(11)-H(11)	119.1(19)
C(3)-P(1)-Pd	109.44(10)	C(13)-C(12)-C(11)	120.3(4)
C(9)-P(1)-Pd	110.06(10)	C(13)-C(12)-H(12)	123(2)
C(2)-P(2)-C(15)	111.07(15)	C(11)-C(12)-H(12)	117(2)
C(2)-P(2)-C(21)	107.16(16)	C(12)-C(13)-C(14)	120.1(4)
C(15)-P(2)-C(21)	104.93(14)	C(12)-C(13)-H(13)	125.7(19)
C(2)-P(2)-Pd	116.48(12)	C(14)-C(13)-H(13)	114.2(19)
C(15)-P(2)-Pd	109.51(11)	C(13)-C(14)-C(9)	120.7(3)
C(21)-P(2)-Pd	106.95(10)	C(13)-C(14)-H(14)	122.5(17)
C(27)-B(1)-C(33)	108.7(2)	C(9)-C(14)-H(14)	116.8(18)
C(27)-B(1)-C(1)	110.4(3)	C(16)-C(15)-C(20)	118.6(3)
C(33)-B(1)-C(1)	108.3(3)	C(16)-C(15)-P(2)	121.5(2)
C(27)-B(1)-C(2)	109.8(3)	C(20)-C(15)-P(2)	119.8(3)
C(33)-B(1)-C(2)	110.1(3)	C(17)-C(16)-C(15)	121.2(4)
C(1)-B(1)-C(2)	109.5(3)	C(17)-C(16)-H(16)	119.3(16)
S-O(2)-Pd	121.90(11)	C(15)-C(16)-H(16)	119.2(15)
B(1)-C(1)-P(1)	120.1(2)	C(18)-C(17)-C(16)	119.7(4)
B(1)-C(1)-H(1A)	111.2(16)	C(18)-C(17)-H(17)	120.8(19)
P(1)-C(1)-H(1A)	100.5(15)	C(16)-C(17)-H(17)	119(2)
B(1)-C(1)-H(1B)	114.7(18)	C(17)-C(18)-C(19)	120.1(4)
P(1)-C(1)-H(1B)	105.2(18)	C(17)-C(18)-H(18)	118.2(19)
H(1A)-C(1)-H(1B)	103(2)	C(19)-C(18)-H(18)	121.7(19)
B(1)-C(2)-P(2)	117.2(2)	C(18)-C(19)-C(20)	120.5(4)

C(18)-C(19)-H(19)	124(2)	C(36)-C(37)-H(37)	125.5(19)
C(20)-C(19)-H(19)	116(2)	C(38)-C(37)-H(37)	113.4(19)
C(19)-C(20)-C(15)	119.8(4)	C(37)-C(38)-C(33)	122.3(4)
C(19)-C(20)-H(20)	121.8(18)	C(37)-C(38)-H(38)	117.7(17)
C(15)-C(20)-H(20)	118.1(18)	C(33)-C(38)-H(38)	120.0(17)
C(22)-C(21)-C(26)	118.6(3)	F(2)-C(39)-F(3)	107.5(3)
C(22)-C(21)-P(2)	119.6(2)	F(2)-C(39)-F(1)	107.7(3)
C(26)-C(21)-P(2)	121.8(3)	F(3)-C(39)-F(1)	108.0(3)
C(21)-C(22)-C(23)	120.5(4)	F(2)-C(39)-S	111.6(2)
C(21)-C(22)-H(22)	123.0(18)	F(3)-C(39)-S	111.3(2)
C(23)-C(22)-H(22)	116.4(18)	F(1)-C(39)-S	110.7(2)
C(24)-C(23)-C(22)	120.1(4)	C(43)-O(1)-C(40)	109.9(2)
C(24)-C(23)-H(23)	118(2)	C(43)-O(1)-Pd	114.92(17)
C(22)-C(23)-H(23)	122(2)	C(40)-O(1)-Pd	133.80(17)
C(23)-C(24)-C(25)	120.2(4)	O(1)-C(40)-C(41)	104.7(2)
C(23)-C(24)-H(24)	119.7(17)	O(1)-C(40)-H(40A)	105.6(17)
C(25)-C(24)-H(24)	120.0(17)	C(41)-C(40)-H(40A)	113.8(17)
C(24)-C(25)-C(26)	120.0(3)	O(1)-C(40)-H(40B)	106.8(15)
C(24)-C(25)-H(25)	121.2(16)	C(41)-C(40)-H(40B)	116.7(15)
C(26)-C(25)-H(25)	118.8(16)	H(40A)-C(40)-H(40B)	108(2)
C(25)-C(26)-C(21)	120.5(3)	C(40)-C(41)-C(42)	101.0(3)
C(25)-C(26)-H(26)	118.1(17)	C(40)-C(41)-H(41A)	110.8(16)
C(21)-C(26)-H(26)	121.4(17)	C(42)-C(41)-H(41A)	107.1(16)
C(28)-C(27)-C(32)	115.2(3)	C(40)-C(41)-H(41B)	115.1(16)
C(28)-C(27)-B(1)	124.3(3)	C(42)-C(41)-H(41B)	110.8(16)
C(32)-C(27)-B(1)	120.5(3)	H(41A)-C(41)-H(41B)	111(2)
C(27)-C(28)-C(29)	122.6(3)	C(43)-C(42)-C(41)	104.6(3)
C(27)-C(28)-H(28)	121.9(17)	C(43)-C(42)-H(42A)	112.4(18)
C(29)-C(28)-H(28)	115.5(17)	C(41)-C(42)-H(42A)	115.3(18)
C(30)-C(29)-C(28)	119.9(4)	C(43)-C(42)-H(42B)	108.4(17)
C(30)-C(29)-H(29)	121.2(17)	C(41)-C(42)-H(42B)	110.1(17)
C(28)-C(29)-H(29)	118.9(17)	H(42A)-C(42)-H(42B)	106(2)
C(29)-C(30)-C(31)	119.2(3)	O(1)-C(43)-C(42)	104.3(3)
C(29)-C(30)-H(30)	123.6(18)	O(1)-C(43)-H(43A)	109.7(19)
C(31)-C(30)-H(30)	117.2(18)	C(42)-C(43)-H(43A)	111.3(19)
C(32)-C(31)-C(30)	120.0(4)	O(1)-C(43)-H(43B)	106.8(16)
C(32)-C(31)-H(31)	115(2)	C(42)-C(43)-H(43B)	113.1(16)
C(30)-C(31)-H(31)	124(2)	H(43A)-C(43)-H(43B)	111(2)
C(31)-C(32)-C(27)	123.1(3)	C(54)-O(5)-C(51)	108.4(5)
C(31)-C(32)-H(32)	118.8(16)	C(52)-C(51)-O(5)	106.3(4)
C(27)-C(32)-H(32)	117.9(16)	C(52)-C(51)-H(51A)	110.5
C(34)-C(33)-C(38)	114.7(3)	O(5)-C(51)-H(51A)	110.5
C(34)-C(33)-B(1)	124.0(3)	C(52)-C(51)-H(51B)	110.5
C(38)-C(33)-B(1)	121.3(3)	O(5)-C(51)-H(51B)	110.5
C(33)-C(34)-C(35)	122.7(4)	H(51A)-C(51)-H(51B)	108.7
C(33)-C(34)-H(34)	121.8(18)	C(51)-C(52)-C(53)	101.3(5)
C(35)-C(34)-H(34)	115.4(18)	C(51)-C(52)-H(52A)	111.5
C(36)-C(35)-C(34)	120.9(4)	C(53)-C(52)-H(52A)	111.5
C(36)-C(35)-H(35)	124(2)	C(51)-C(52)-H(52B)	111.5
C(34)-C(35)-H(35)	116(2)	C(53)-C(52)-H(52B)	111.5
C(35)-C(36)-C(37)	118.6(4)	H(52A)-C(52)-H(52B)	109.3
C(35)-C(36)-H(36)	118.9(18)	C(54)-C(53)-C(52)	114.2(4)
C(37)-C(36)-H(36)	122.3(18)	C(54)-C(53)-H(53A)	108.7
C(36)-C(37)-C(38)	120.7(4)	C(52)-C(53)-H(53A)	108.7

C(54)-C(53)-H(53B)	108.7	C(53)-C(54)-H(54A)	110.5
C(52)-C(53)-H(53B)	108.7	O(5)-C(54)-H(54B)	110.5
H(53A)-C(53)-H(53B)	107.6	C(53)-C(54)-H(54B)	110.5
O(5)-C(54)-C(53)	106.0(5)	H(54A)-C(54)-H(54B)	108.7
O(5)-C(54)-H(54A)	110.5		

Table 4. Anisotropic displacement parameters ($Å^2 x 10^4$) for 1' (CCDC 171286). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd	174(1)	252(1)	206(1)	45(1)	38(1)	0(1)
S	242(5)	311(5)	247(5)	-23(4)	38(4)	-14(4)
P(1)	188(4)	257(5)	218(5)	38(4)	50(4)	25(4)
P(2)	186(5)	292(5)	238(5)	68(4)	24(4)	-9(4)
B(1)	174(19)	300(20)	280(20)	72(17)	35(16)	7(16)
F(1)	363(12)	571(14)	305(11)	125(10)	2(9)	67(10)
F(2)	394(12)	425(13)	500(13)	-174(11)	-61(10)	21(10)
F(3)	216(11)	522(13)	444(12)	43(10)	41(9)	-32(9)
O(2)	219(12)	305(13)	196(12)	4(10)	73(9)	-31(10)
O(3)	334(13)	298(13)	348(13)	-99(11)	46(10)	75(11)
O(4)	340(14)	320(13)	233(12)	104(10)	70(10)	2(11)
C(1)	218(19)	310(20)	260(20)	91(17)	106(16)	65(16)
C(2)	210(20)	300(20)	290(20)	63(17)	-6(16)	-48(17)
C(3)	215(17)	241(19)	215(17)	26(14)	10(14)	1(14)
C(4)	380(20)	340(20)	310(20)	96(18)	104(17)	132(18)
C(5)	400(20)	460(30)	500(30)	170(20)	230(20)	230(20)
C(6)	330(20)	380(20)	460(30)	171(19)	63(19)	186(19)
C(7)	310(20)	410(20)	360(20)	178(18)	33(18)	70(18)
C(8)	221(19)	390(20)	290(20)	80(16)	49(16)	78(16)
C(9)	260(19)	251(19)	217(18)	38(14)	104(15)	15(15)
C(10)	320(20)	350(20)	350(20)	-87(18)	67(18)	59(18)
C(11)	380(30)	440(30)	400(20)	-140(19)	30(20)	-10(20)
C(12)	580(30)	330(20)	340(20)	-123(19)	140(20)	-10(20)
C(13)	440(30)	290(20)	390(20)	18(18)	190(20)	100(20)
C(14)	300(20)	330(20)	310(20)	86(17)	112(17)	46(18)
C(15)	193(18)	380(20)	233(18)	106(15)	40(14)	-22(15)
C(16)	240(20)	420(20)	233(19)	62(17)	43(16)	14(18)
C(17)	370(20)	450(30)	310(20)	110(20)	103(18)	150(20)
C(18)	310(20)	650(30)	450(30)	240(20)	110(20)	150(20)
C(19)	210(20)	710(40)	460(30)	250(20)	-39(18)	-90(20)
C(20)	280(20)	420(30)	410(20)	140(20)	17(18)	-43(19)
C(21)	200(17)	310(20)	269(18)	31(16)	34(14)	-77(16)
C(22)	410(20)	510(30)	260(20)	80(20)	22(18)	80(20)
C(23)	520(30)	490(30)	420(30)	-10(20)	60(20)	200(20)
C(24)	420(20)	500(30)	290(20)	-90(20)	39(19)	-50(20)
C(25)	320(20)	430(20)	231(19)	-11(19)	-55(16)	-99(19)
C(26)	250(20)	280(20)	320(20)	18(17)	-27(16)	-90(16)
C(27)	154(17)	300(20)	278(19)	55(15)	41(14)	-39(15)
C(28)	270(20)	360(20)	260(20)	52(17)	33(16)	-20(16)
C(29)	310(20)	300(20)	380(20)	77(18)	60(17)	43(17)
C(30)	300(20)	410(20)	300(20)	187(19)	-4(17)	-3(18)
C(31)	280(20)	470(30)	250(20)	60(19)	81(17)	-41(18)
C(32)	227(19)	300(20)	320(20)	46(18)	88(15)	11(16)
C(33)	245(18)	320(20)	263(18)	157(17)	64(14)	-3(17)
C(34)	250(20)	460(30)	360(20)	82(19)	63(18)	8(19)
C(35)	250(20)	540(30)	550(30)	140(20)	130(20)	90(20)
C(36)	190(20)	570(30)	600(30)	300(20)	30(20)	0(20)

C(37)	310(20)	480(30)	520(30)	230(20)	-40(20)	-170(20)
C(38)	310(20)	320(20)	530(30)	140(20)	48(19)	-27(19)
C(39)	330(20)	310(20)	260(20)	25(16)	39(16)	45(17)
O(1)	261(12)	244(12)	199(12)	46(9)	62(9)	46(10)
C(40)	233(19)	310(20)	239(18)	-16(16)	68(15)	0(17)
C(41)	340(20)	320(20)	260(20)	57(16)	85(18)	12(17)
C(42)	320(20)	310(20)	350(20)	94(18)	74(18)	55(19)
C(43)	320(20)	260(20)	300(20)	68(16)	47(18)	56(17)
O(5)	1050(40)	1310(40)	2660(70)	1090(40)	-540(40)	-360(30)
C(51)	1620(80)	3660(140)	880(50)	910(70)	860(60)	630(90)
C(52)	790(50)	2130(90)	1960(80)	1450(70)	120(50)	-110(50)
C(53)	890(50)	500(40)	2240(80)	330(40)	310(50)	-90(30)
C(54)	1480(70)	1360(60)	820(40)	540(40)	-220(40)	-690(50)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for 1' (CCDC 171286).

	x	y	z	U_{iso}
H(1A)	7019(18)	2023(15)	8299(12)	14(7)
H(1B)	6630(20)	2388(16)	7638(15)	34(9)
H(2A)	6140(20)	3702(15)	9090(13)	23(8)
H(2B)	7190(20)	3815(15)	9422(13)	17(8)
H(4)	3680(20)	2873(17)	8876(16)	47(10)
H(5)	2470(20)	3752(18)	8396(16)	42(11)
H(6)	2620(20)	4340(17)	7443(16)	35(10)
H(7)	3930(20)	4143(17)	6730(16)	44(10)
H(8)	5130(20)	3219(15)	7160(14)	28(9)
H(10)	3620(20)	1685(15)	7525(14)	20(9)
H(11)	3190(20)	700(16)	6797(14)	24(9)
H(12)	4340(20)	-187(18)	6606(16)	42(11)
H(13)	5990(20)	-91(16)	7173(14)	28(9)
H(14)	6410(20)	891(16)	7912(14)	27(9)
H(16)	7171(19)	1142(14)	9546(13)	13(7)
H(17)	8610(20)	425(17)	9869(15)	31(10)
H(18)	10080(20)	976(18)	10356(15)	44(10)
H(19)	10020(20)	2301(17)	10617(15)	32(10)
H(20)	8590(20)	2987(16)	10388(13)	19(9)
H(22)	5600(20)	4082(16)	10196(15)	29(9)
H(23)	5350(20)	4575(18)	11255(16)	44(12)
H(24)	5900(20)	4055(15)	12292(14)	26(8)
H(25)	6775(19)	2878(15)	12294(14)	23(8)
H(26)	7090(20)	2348(15)	11249(13)	18(8)
H(28)	6420(20)	4778(16)	8505(15)	36(9)
H(29)	6130(20)	5710(16)	7669(14)	23(8)
H(30)	6490(20)	5469(16)	6506(14)	25(9)
H(31)	7140(20)	4304(16)	6259(15)	23(9)
H(32)	7430(18)	3406(14)	7037(12)	6(7)
H(34)	8560(20)	2111(16)	7999(14)	25(9)
H(35)	10180(20)	1906(17)	8209(16)	38(11)
H(36)	11260(20)	2702(16)	8921(15)	35(9)
H(37)	10470(20)	3835(17)	9378(16)	37(10)
H(38)	8835(19)	4038(14)	9108(13)	9(8)
H(40A)	3950(20)	1781(15)	10937(14)	29(9)
H(40B)	4780(20)	2416(16)	11166(13)	25(9)
H(41A)	5670(20)	1523(16)	11963(14)	33(9)
H(41B)	4480(20)	1260(15)	12067(14)	29(8)
H(42A)	5570(20)	252(16)	11613(14)	24(9)
H(42B)	4540(20)	340(16)	11203(14)	28(9)
H(43A)	6360(20)	885(17)	10806(16)	42(10)
H(43B)	5460(20)	480(16)	10278(15)	39(9)
H(51A)	5824	1012	5628	240
H(51B)	6603	599	6206	240
H(52A)	7311	1732	6348	195
H(52B)	6789	2035	5603	195
H(53A)	8247	1813	5219	144

H(53B)	8626	1308	5889	144
H(54A)	8013	825	4604	149
H(54B)	8469	324	5265	149

Figure 2. Labeled drawing of 5 (CCDC 172948), with 50% ellipsoids. Hydrogen atoms and solvent molecules have been omitted for clarity.

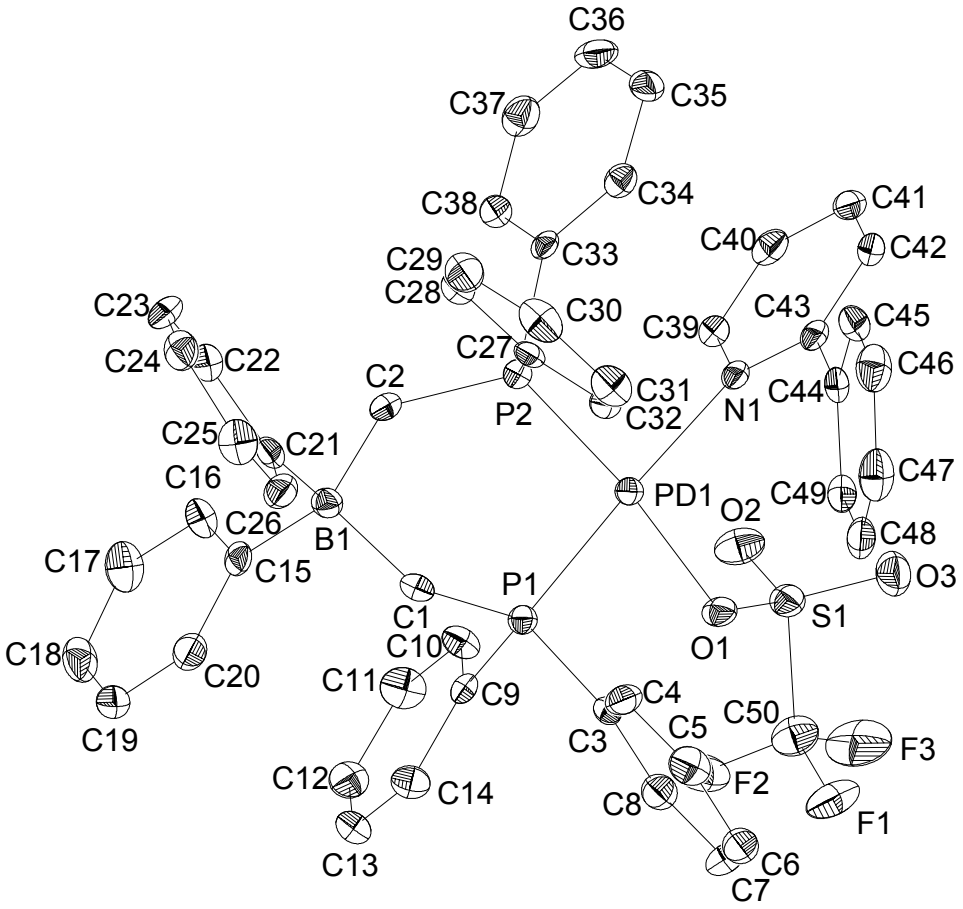

Table 6. Crystal data and structure refinement for 5 (CCDC 172948).

Empirical formula	$C_{50}H_{43}BF_3NO_3P_2PdS \cdot 2(CH_2Cl_2)$
Formula weight	1143.92
Crystallization Solvent	Dichloromethane/petroleum ether
Crystal Habit	Plate
Crystal size	0.29 x 0.28 x 0.05 mm^3
Crystal color	Dichroic Pink/Yellow

Data Collection

Preliminary Photos	Rotation	
Type of diffractometer	CCD area detector	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	98(2) K	
θ range for 25758 reflections used in lattice determination	2.75 to 25.50°	
Unit cell dimensions	a = 16.3584(11) Å b = 17.4150(12) Å c = 19.5418(13) Å	β= 112.1880(10)°
Volume	5154.9(6) $Å^3$	
Z	4	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Density (calculated)	1.474 Mg/m^3	
F(000)	2328	
Data collection program	Bruker SMART	
θ range for data collection	1.39 to 28.42°	
Completeness to θ = 28.42°	94.8 %	
Index ranges	$-21 \le h \le 21$, $-23 \le k \le 23$, $-25 \le l \le 25$	
Data collection scan type	ω scans at 7 ϕ settings	
Data reduction program	Bruker SAINT v6.2	
Reflections collected	104619	
Independent reflections	12279 [R_{int}= 0.0830]	
Absorption coefficient	0.724 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9661 and 0.8186	

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Patterson method
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	12279 / 0 / 785
Treatment of hydrogen atoms	Unrestrained except for solvent
Goodness-of-fit on F^2	1.940
Final R indices [$I>2\sigma(I)$, 8471 reflections]	R1 = 0.0489, *w*R2 = 0.0802
R indices (all data)	R1 = 0.0820, *w*R2 = 0.0830
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.106
Average shift/error	0.000
Largest diff. peak and hole	1.648 and -1.247 e.Å^{-3}

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 7. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 5 (CCDC 172948). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Pd(1)	5298(1)	1351(1)	7735(1)	15(1)
S(1)	4153(1)	-254(1)	7139(1)	21(1)
P(1)	4770(1)	2217(1)	6801(1)	15(1)
P(2)	6489(1)	2108(1)	8250(1)	16(1)
F(1)	2516(1)	-92(1)	6187(1)	39(1)
F(2)	3469(1)	-263(1)	5688(1)	40(1)
F(3)	3079(2)	-1217(1)	6201(1)	52(1)
N(1)	5737(2)	518(2)	8603(1)	15(1)
O(1)	4207(1)	588(1)	7099(1)	20(1)
O(2)	4899(2)	-654(1)	7095(1)	30(1)
O(3)	3824(2)	-511(1)	7685(1)	33(1)
B(1)	6372(3)	3141(2)	7001(2)	17(1)
C(1)	5305(2)	3128(2)	6884(2)	17(1)
C(2)	6906(2)	2423(2)	7573(2)	18(1)
C(3)	3633(2)	2401(2)	6702(2)	15(1)
C(4)	3439(2)	3008(2)	7089(2)	21(1)
C(5)	2589(3)	3148(2)	7037(2)	26(1)
C(6)	1902(3)	2690(2)	6594(2)	27(1)
C(7)	2074(3)	2091(2)	6203(2)	27(1)
C(8)	2932(2)	1944(2)	6258(2)	22(1)
C(9)	4751(2)	1803(2)	5945(2)	15(1)
C(10)	5267(2)	1165(2)	5960(2)	21(1)
C(11)	5353(3)	911(2)	5314(2)	27(1)
C(12)	4924(2)	1290(2)	4653(2)	24(1)
C(13)	4394(2)	1920(2)	4633(2)	23(1)
C(14)	4309(2)	2176(2)	5275(2)	20(1)
C(15)	6512(2)	3057(2)	6218(2)	17(1)
C(16)	7129(3)	2571(2)	6105(2)	23(1)
C(17)	7243(3)	2517(2)	5433(2)	30(1)
C(18)	6734(3)	2957(2)	4835(2)	29(1)
C(19)	6123(3)	3452(2)	4923(2)	27(1)
C(20)	6022(2)	3501(2)	5602(2)	23(1)
C(21)	6786(2)	3964(2)	7383(2)	17(1)
C(22)	7706(2)	4082(2)	7695(2)	21(1)
C(23)	8084(3)	4742(2)	8079(2)	24(1)
C(24)	7567(3)	5336(2)	8154(2)	24(1)
C(25)	6656(3)	5265(2)	7822(2)	26(1)
C(26)	6283(3)	4595(2)	7445(2)	22(1)
C(27)	6315(2)	2926(2)	8752(2)	16(1)
C(28)	7004(3)	3459(2)	9050(2)	22(1)
C(29)	6886(3)	4099(2)	9430(2)	28(1)
C(30)	6106(3)	4207(2)	9526(2)	29(1)
C(31)	5425(3)	3691(2)	9241(2)	26(1)
C(32)	5528(2)	3054(2)	8852(2)	20(1)
C(33)	7363(2)	1567(2)	8954(2)	15(1)
C(34)	7390(2)	1519(2)	9677(2)	19(1)
C(35)	8016(3)	1065(2)	10198(2)	24(1)

C(36)	8626(3)	665(2)	10004(2)	27(1)
C(37)	8613(3)	713(2)	9295(2)	26(1)
C(38)	7981(2)	1157(2)	8772(2)	22(1)
C(39)	6308(2)	-23(2)	8558(2)	19(1)
C(40)	6717(2)	-548(2)	9121(2)	22(1)
C(41)	6546(2)	-497(2)	9763(2)	22(1)
C(42)	5964(2)	63(2)	9812(2)	20(1)
C(43)	5555(2)	559(2)	9224(2)	17(1)
C(44)	4924(2)	1156(2)	9259(2)	19(1)
C(45)	5112(3)	1601(2)	9897(2)	24(1)
C(46)	4537(3)	2167(2)	9935(2)	28(1)
C(47)	3764(3)	2297(2)	9341(2)	29(1)
C(48)	3549(3)	1861(2)	8707(2)	24(1)
C(49)	4122(2)	1288(2)	8664(2)	20(1)
C(50)	3261(3)	-466(2)	6256(2)	30(1)
Cl(1)	5361(1)	7363(1)	6968(1)	69(1)
Cl(2)	5166(1)	6815(1)	8296(1)	51(1)
C(51)	5350(3)	7629(2)	7826(2)	49(1)
Cl(3)	916(1)	-46(1)	6878(1)	53(1)
Cl(4)	1633(1)	513(1)	8389(1)	67(1)
C(52)	1777(3)	476(2)	7541(2)	45(1)

Table 8. Bond lengths [Å] and angles [°] for 5 (CCDC 172948).

Pd(1)-N(1)	2.139(3)	C(15)-C(16)	1.398(5)
Pd(1)-O(1)	2.195(2)	C(16)-C(17)	1.397(5)
Pd(1)-P(2)	2.2503(9)	C(16)-H(16)	0.91(3)
Pd(1)-P(1)	2.2718(9)	C(17)-C(18)	1.383(5)
S(1)-O(3)	1.436(2)	C(17)-H(17)	0.86(3)
S(1)-O(2)	1.436(2)	C(18)-C(19)	1.380(5)
S(1)-O(1)	1.474(2)	C(18)-H(18)	0.93(3)
S(1)-C(50)	1.828(4)	C(19)-C(20)	1.399(5)
P(1)-C(1)	1.790(4)	C(19)-H(19)	0.84(3)
P(1)-C(9)	1.811(3)	C(20)-H(20)	0.94(3)
P(1)-C(3)	1.824(3)	C(21)-C(22)	1.410(5)
P(2)-C(2)	1.789(4)	C(21)-C(26)	1.405(5)
P(2)-C(27)	1.812(3)	C(22)-C(23)	1.383(5)
P(2)-C(33)	1.826(3)	C(22)-H(22)	0.94(3)
F(1)-C(50)	1.343(4)	C(23)-C(24)	1.379(5)
F(2)-C(50)	1.325(4)	C(23)-H(23)	0.81(3)
F(3)-C(50)	1.335(4)	C(24)-C(25)	1.388(5)
N(1)-C(43)	1.355(4)	C(24)-H(24)	0.99(3)
N(1)-C(39)	1.353(4)	C(25)-C(26)	1.392(5)
B(1)-C(15)	1.636(5)	C(25)-H(25)	0.97(3)
B(1)-C(21)	1.640(5)	C(26)-H(26)	0.87(3)
B(1)-C(1)	1.672(5)	C(27)-C(32)	1.393(5)
B(1)-C(2)	1.685(5)	C(27)-C(28)	1.406(5)
C(1)-H(1A)	0.89(3)	C(28)-C(29)	1.393(5)
C(1)-H(1B)	0.94(3)	C(28)-H(28)	0.95(3)
C(2)-H(2A)	0.95(3)	C(29)-C(30)	1.370(5)
C(2)-H(2B)	0.92(3)	C(29)-H(29)	0.83(3)
C(3)-C(8)	1.396(5)	C(30)-C(31)	1.375(5)
C(3)-C(4)	1.404(5)	C(30)-H(30)	0.86(3)
C(4)-C(5)	1.378(5)	C(31)-C(32)	1.390(5)
C(4)-H(4)	0.83(3)	C(31)-H(31)	0.90(3)
C(5)-C(6)	1.383(5)	C(32)-H(32)	0.83(3)
C(5)-H(5)	0.86(3)	C(33)-C(38)	1.390(5)
C(6)-C(7)	1.384(5)	C(33)-C(34)	1.399(5)
C(6)-H(6)	0.92(3)	C(34)-C(35)	1.387(5)
C(7)-C(8)	1.391(5)	C(34)-H(34)	0.91(3)
C(7)-H(7)	0.88(3)	C(35)-C(36)	1.381(5)
C(8)-H(8)	0.91(3)	C(35)-H(35)	0.79(3)
C(9)-C(10)	1.389(4)	C(36)-C(37)	1.381(5)
C(9)-C(14)	1.395(4)	C(36)-H(36)	0.84(3)
C(10)-C(11)	1.394(5)	C(37)-C(38)	1.383(5)
C(10)-H(10)	0.88(3)	C(37)-H(37)	0.84(3)
C(11)-C(12)	1.384(5)	C(38)-H(38)	0.86(3)
C(11)-H(11)	0.93(3)	C(39)-C(40)	1.392(5)
C(12)-C(13)	1.390(5)	C(39)-H(39)	0.91(3)
C(12)-H(12)	0.95(3)	C(40)-C(41)	1.389(5)
C(13)-C(14)	1.385(5)	C(40)-H(40)	0.93(3)
C(13)-H(13)	0.89(3)	C(41)-C(42)	1.390(5)
C(14)-H(14)	0.92(3)	C(41)-H(41)	0.93(3)
C(15)-C(20)	1.401(4)	C(42)-C(43)	1.391(4)

C(42)-H(42)	0.95(3)	C(21)-B(1)-C(2)	109.0(3)
C(43)-C(44)	1.486(4)	C(1)-B(1)-C(2)	109.5(3)
C(44)-C(45)	1.399(5)	B(1)-C(1)-P(1)	118.2(2)
C(44)-C(49)	1.404(5)	B(1)-C(1)-H(1A)	111.7(18)
C(45)-C(46)	1.385(5)	P(1)-C(1)-H(1A)	104.3(18)
C(45)-H(45)	0.89(3)	B(1)-C(1)-H(1B)	111.9(17)
C(46)-C(47)	1.375(6)	P(1)-C(1)-H(1B)	102.6(16)
C(46)-H(46)	0.91(3)	H(1A)-C(1)-H(1B)	107(2)
C(47)-C(48)	1.381(5)	B(1)-C(2)-P(2)	118.1(2)
C(47)-H(47)	0.77(3)	B(1)-C(2)-H(2A)	112.4(18)
C(48)-C(49)	1.393(5)	P(2)-C(2)-H(2A)	102.8(18)
C(48)-H(48)	1.02(3)	B(1)-C(2)-H(2B)	110.0(19)
C(49)-H(49)	0.98(3)	P(2)-C(2)-H(2B)	108.7(19)
Cl(1)-C(51)	1.746(4)	H(2A)-C(2)-H(2B)	104(3)
Cl(2)-C(51)	1.776(4)	C(8)-C(3)-C(4)	117.8(3)
C(51)-H(51A)	0.9900	C(8)-C(3)-P(1)	122.0(3)
C(51)-H(51B)	0.9900	C(4)-C(3)-P(1)	120.2(3)
Cl(3)-C(52)	1.764(4)	C(5)-C(4)-C(3)	121.4(4)
Cl(4)-C(52)	1.761(4)	C(5)-C(4)-H(4)	120(2)
C(52)-H(52A)	0.9900	C(3)-C(4)-H(4)	119(2)
C(52)-H(52B)	0.9900	C(4)-C(5)-C(6)	120.2(4)
		C(4)-C(5)-H(5)	120(2)
N(1)-Pd(1)-O(1)	89.85(9)	C(6)-C(5)-H(5)	120(2)
N(1)-Pd(1)-P(2)	93.87(7)	C(7)-C(6)-C(5)	119.7(4)
O(1)-Pd(1)-P(2)	171.45(6)	C(7)-C(6)-H(6)	122(2)
N(1)-Pd(1)-P(1)	177.49(7)	C(5)-C(6)-H(6)	118(2)
O(1)-Pd(1)-P(1)	88.00(6)	C(6)-C(7)-C(8)	120.4(4)
P(2)-Pd(1)-P(1)	88.43(3)	C(6)-C(7)-H(7)	118(2)
O(3)-S(1)-O(2)	117.33(15)	C(8)-C(7)-H(7)	122(2)
O(3)-S(1)-O(1)	113.40(15)	C(3)-C(8)-C(7)	120.7(4)
O(2)-S(1)-O(1)	114.16(14)	C(3)-C(8)-H(8)	117.1(19)
O(3)-S(1)-C(50)	104.44(17)	C(7)-C(8)-H(8)	121.7(19)
O(2)-S(1)-C(50)	103.84(16)	C(10)-C(9)-C(14)	119.1(3)
O(1)-S(1)-C(50)	101.15(15)	C(10)-C(9)-P(1)	120.1(3)
C(1)-P(1)-C(9)	105.88(16)	C(14)-C(9)-P(1)	120.3(3)
C(1)-P(1)-C(3)	107.37(16)	C(9)-C(10)-C(11)	120.3(3)
C(9)-P(1)-C(3)	108.08(15)	C(9)-C(10)-H(10)	117(2)
C(1)-P(1)-Pd(1)	119.44(12)	C(11)-C(10)-H(10)	123(2)
C(9)-P(1)-Pd(1)	110.25(11)	C(12)-C(11)-C(10)	120.3(4)
C(3)-P(1)-Pd(1)	105.38(10)	C(12)-C(11)-H(11)	119(2)
C(2)-P(2)-C(27)	109.88(16)	C(10)-C(11)-H(11)	121(2)
C(2)-P(2)-C(33)	107.81(16)	C(11)-C(12)-C(13)	119.6(3)
C(27)-P(2)-C(33)	103.63(15)	C(11)-C(12)-H(12)	123.3(19)
C(2)-P(2)-Pd(1)	110.91(13)	C(13)-C(12)-H(12)	117.1(18)
C(27)-P(2)-Pd(1)	114.64(12)	C(14)-C(13)-C(12)	120.2(4)
C(33)-P(2)-Pd(1)	109.53(10)	C(14)-C(13)-H(13)	118(2)
C(43)-N(1)-C(39)	119.0(3)	C(12)-C(13)-H(13)	121(2)
C(43)-N(1)-Pd(1)	123.6(2)	C(13)-C(14)-C(9)	120.5(3)
C(39)-N(1)-Pd(1)	117.0(2)	C(13)-C(14)-H(14)	119.1(17)
S(1)-O(1)-Pd(1)	128.63(13)	C(9)-C(14)-H(14)	120.3(17)
C(15)-B(1)-C(21)	108.9(3)	C(20)-C(15)-C(16)	114.2(3)
C(15)-B(1)-C(1)	112.2(3)	C(20)-C(15)-B(1)	121.2(3)
C(21)-B(1)-C(1)	107.7(3)	C(16)-C(15)-B(1)	124.5(3)
C(15)-B(1)-C(2)	109.5(3)	C(15)-C(16)-C(17)	123.4(4)

C(15)-C(16)-H(16)	118(2)	C(35)-C(34)-H(34)	118.9(19)
C(17)-C(16)-H(16)	118(2)	C(33)-C(34)-H(34)	120.4(19)
C(18)-C(17)-C(16)	120.4(4)	C(36)-C(35)-C(34)	119.7(4)
C(18)-C(17)-H(17)	120(2)	C(36)-C(35)-H(35)	125(3)
C(16)-C(17)-H(17)	120(2)	C(34)-C(35)-H(35)	115(3)
C(19)-C(18)-C(17)	118.2(4)	C(35)-C(36)-C(37)	120.3(4)
C(19)-C(18)-H(18)	119(2)	C(35)-C(36)-H(36)	115(2)
C(17)-C(18)-H(18)	123(2)	C(37)-C(36)-H(36)	125(2)
C(18)-C(19)-C(20)	120.5(4)	C(38)-C(37)-C(36)	120.0(4)
C(18)-C(19)-H(19)	119(2)	C(38)-C(37)-H(37)	116(2)
C(20)-C(19)-H(19)	120(2)	C(36)-C(37)-H(37)	124(2)
C(15)-C(20)-C(19)	123.2(4)	C(33)-C(38)-C(37)	120.8(4)
C(15)-C(20)-H(20)	116.2(19)	C(33)-C(38)-H(38)	116(2)
C(19)-C(20)-H(20)	121(2)	C(37)-C(38)-H(38)	123(2)
C(22)-C(21)-C(26)	114.4(3)	N(1)-C(39)-C(40)	123.0(3)
C(22)-C(21)-B(1)	121.0(3)	N(1)-C(39)-H(39)	119.2(18)
C(26)-C(21)-B(1)	124.6(3)	C(40)-C(39)-H(39)	117.9(18)
C(23)-C(22)-C(21)	122.8(4)	C(39)-C(40)-C(41)	118.1(4)
C(23)-C(22)-H(22)	115.1(19)	C(39)-C(40)-H(40)	117(2)
C(21)-C(22)-H(22)	122.0(19)	C(41)-C(40)-H(40)	125(2)
C(22)-C(23)-C(24)	121.0(4)	C(42)-C(41)-C(40)	118.9(3)
C(22)-C(23)-H(23)	115(2)	C(42)-C(41)-H(41)	124(2)
C(24)-C(23)-H(23)	124(2)	C(40)-C(41)-H(41)	117(2)
C(25)-C(24)-C(23)	118.4(4)	C(43)-C(42)-C(41)	120.5(3)
C(25)-C(24)-H(24)	121.0(18)	C(43)-C(42)-H(42)	119.4(17)
C(23)-C(24)-H(24)	120.6(18)	C(41)-C(42)-H(42)	120.0(17)
C(26)-C(25)-C(24)	120.1(4)	N(1)-C(43)-C(42)	120.4(3)
C(26)-C(25)-H(25)	118(2)	N(1)-C(43)-C(44)	118.0(3)
C(24)-C(25)-H(25)	121(2)	C(42)-C(43)-C(44)	121.5(3)
C(25)-C(26)-C(21)	123.2(4)	C(45)-C(44)-C(49)	118.0(3)
C(25)-C(26)-H(26)	117(2)	C(45)-C(44)-C(43)	120.4(3)
C(21)-C(26)-H(26)	120(2)	C(49)-C(44)-C(43)	121.6(3)
C(32)-C(27)-C(28)	118.4(3)	C(46)-C(45)-C(44)	121.2(4)
C(32)-C(27)-P(2)	123.0(3)	C(46)-C(45)-H(45)	119(2)
C(28)-C(27)-P(2)	118.6(3)	C(44)-C(45)-H(45)	120(2)
C(29)-C(28)-C(27)	119.9(4)	C(45)-C(46)-C(47)	119.9(4)
C(29)-C(28)-H(28)	122.4(19)	C(45)-C(46)-H(46)	121(2)
C(27)-C(28)-H(28)	117.7(19)	C(47)-C(46)-H(46)	119(2)
C(30)-C(29)-C(28)	120.5(4)	C(48)-C(47)-C(46)	120.6(4)
C(30)-C(29)-H(29)	120(2)	C(48)-C(47)-H(47)	120(3)
C(28)-C(29)-H(29)	119(2)	C(46)-C(47)-H(47)	120(3)
C(29)-C(30)-C(31)	120.5(4)	C(47)-C(48)-C(49)	119.9(4)
C(29)-C(30)-H(30)	118(2)	C(47)-C(48)-H(48)	120.8(18)
C(31)-C(30)-H(30)	120(2)	C(49)-C(48)-H(48)	119.2(18)
C(30)-C(31)-C(32)	119.8(4)	C(48)-C(49)-C(44)	120.4(3)
C(30)-C(31)-H(31)	120(2)	C(48)-C(49)-H(49)	121.3(17)
C(32)-C(31)-H(31)	120(2)	C(44)-C(49)-H(49)	118.3(17)
C(27)-C(32)-C(31)	120.9(4)	F(2)-C(50)-F(3)	108.3(3)
C(27)-C(32)-H(32)	120(2)	F(2)-C(50)-F(1)	107.3(3)
C(31)-C(32)-H(32)	119(2)	F(3)-C(50)-F(1)	107.5(3)
C(38)-C(33)-C(34)	118.5(3)	F(2)-C(50)-S(1)	111.7(3)
C(38)-C(33)-P(2)	120.7(3)	F(3)-C(50)-S(1)	110.3(3)
C(34)-C(33)-P(2)	120.6(3)	F(1)-C(50)-S(1)	111.5(3)
C(35)-C(34)-C(33)	120.6(4)	Cl(1)-C(51)-Cl(2)	110.5(2)

Cl(1)-C(51)-H(51A)	109.5
Cl(2)-C(51)-H(51A)	109.5
Cl(1)-C(51)-H(51B)	109.5
Cl(2)-C(51)-H(51B)	109.5
H(51A)-C(51)-H(51B)	108.1
Cl(4)-C(52)-Cl(3)	110.9(2)
Cl(4)-C(52)-H(52A)	109.5
Cl(3)-C(52)-H(52A)	109.5
Cl(4)-C(52)-H(52B)	109.5
Cl(3)-C(52)-H(52B)	109.5
H(52A)-C(52)-H(52B)	108.0

Table 9. Anisotropic displacement parameters ($Å^2 x 10^4$) for 5 (CCDC 172948). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd(1)	142(1)	142(1)	145(1)	12(1)	41(1)	-12(1)
S(1)	217(5)	190(5)	191(5)	-3(4)	37(4)	-43(4)
P(1)	144(5)	140(5)	150(5)	4(4)	49(4)	-5(4)
P(2)	145(5)	166(5)	153(5)	10(4)	50(4)	-16(4)
F(1)	181(12)	515(15)	411(14)	-101(12)	33(11)	-14(11)
F(2)	380(14)	571(16)	204(12)	-34(11)	57(11)	88(12)
F(3)	526(16)	265(14)	529(16)	-127(12)	-65(13)	-131(12)
N(1)	122(15)	146(15)	173(16)	10(12)	40(13)	-29(12)
O(1)	180(13)	148(13)	207(13)	17(11)	4(11)	-44(10)
O(2)	230(15)	277(15)	334(16)	-49(12)	33(12)	47(12)
O(3)	378(17)	333(16)	271(15)	7(12)	130(13)	-135(13)
B(1)	180(20)	150(20)	160(20)	14(17)	48(18)	-7(17)
C(1)	190(20)	168(19)	117(19)	9(16)	32(17)	21(16)
C(2)	110(20)	210(20)	180(20)	11(16)	29(17)	-16(16)
C(3)	148(18)	141(18)	142(18)	48(14)	48(15)	19(15)
C(4)	150(20)	250(20)	210(20)	-11(17)	31(17)	-11(17)
C(5)	270(20)	300(20)	230(20)	3(18)	117(19)	70(19)
C(6)	170(20)	380(20)	270(20)	103(19)	103(19)	77(19)
C(7)	150(20)	360(20)	280(20)	2(19)	34(19)	-47(19)
C(8)	220(20)	240(20)	210(20)	13(17)	87(18)	19(17)
C(9)	110(18)	165(18)	177(19)	-9(15)	61(15)	-23(14)
C(10)	250(20)	220(20)	170(20)	36(16)	63(18)	54(17)
C(11)	290(20)	270(20)	280(20)	-38(18)	130(20)	97(19)
C(12)	250(20)	300(20)	170(20)	-66(18)	90(17)	-4(18)
C(13)	240(20)	280(20)	160(20)	45(17)	57(18)	-2(18)
C(14)	200(20)	170(20)	220(20)	14(16)	61(17)	63(17)
C(15)	184(19)	140(18)	190(20)	-10(15)	75(16)	-66(15)
C(16)	290(20)	210(20)	220(20)	52(17)	125(19)	2(18)
C(17)	390(30)	250(20)	350(30)	-20(19)	240(20)	10(20)
C(18)	370(30)	330(20)	210(20)	-50(19)	170(20)	-90(20)
C(19)	230(20)	330(30)	200(20)	48(18)	37(19)	-81(18)
C(20)	200(20)	250(20)	250(20)	-6(17)	95(18)	-28(17)
C(21)	210(20)	195(19)	123(18)	29(15)	70(16)	-6(16)
C(22)	230(20)	200(20)	220(20)	20(16)	106(18)	-8(17)
C(23)	140(20)	280(20)	260(20)	1(18)	35(18)	-68(18)
C(24)	300(20)	200(20)	230(20)	-24(17)	97(19)	-91(18)
C(25)	330(20)	150(20)	320(20)	-7(17)	170(20)	39(18)
C(26)	180(20)	210(20)	260(20)	21(17)	66(18)	-27(17)
C(27)	175(19)	178(19)	106(18)	38(15)	23(15)	4(15)
C(28)	220(20)	270(20)	160(20)	19(16)	64(17)	-19(17)
C(29)	350(30)	250(20)	210(20)	-42(18)	60(20)	-130(20)
C(30)	440(30)	200(20)	220(20)	-9(18)	130(20)	20(20)
C(31)	310(20)	250(20)	250(20)	21(18)	127(19)	60(20)
C(32)	190(20)	210(20)	170(20)	22(16)	41(17)	-34(17)
C(33)	113(18)	135(18)	179(19)	-4(14)	33(15)	-41(14)
C(34)	170(20)	170(20)	220(20)	-12(15)	71(17)	-23(16)
C(35)	250(20)	250(20)	160(20)	19(17)	13(19)	-89(17)
C(36)	180(20)	220(20)	300(20)	63(19)	-41(19)	-11(18)
C(37)	210(20)	220(20)	360(30)	38(19)	110(20)	17(18)

C(38)	200(20)	270(20)	180(20)	19(17)	50(18)	-61(16)
C(39)	160(20)	220(20)	180(20)	10(16)	58(17)	-22(16)
C(40)	160(20)	170(20)	300(20)	-13(17)	67(18)	-20(16)
C(41)	160(20)	220(20)	240(20)	76(17)	9(17)	-34(17)
C(42)	170(20)	260(20)	180(20)	23(17)	72(17)	-57(16)
C(43)	130(19)	168(19)	195(19)	6(15)	45(16)	-47(15)
C(44)	200(20)	200(20)	190(20)	36(15)	104(17)	-38(15)
C(45)	250(20)	300(20)	170(20)	30(17)	84(19)	-50(18)
C(46)	350(30)	290(20)	280(20)	-66(19)	210(20)	-52(19)
C(47)	340(30)	210(20)	440(30)	20(20)	280(20)	50(20)
C(48)	230(20)	240(20)	300(20)	55(18)	150(20)	-15(18)
C(49)	220(20)	200(20)	210(20)	-5(17)	119(17)	-36(17)
C(50)	280(20)	250(20)	320(20)	-69(19)	70(20)	-19(19)
Cl(1)	1052(12)	681(9)	459(8)	-80(7)	437(8)	-262(8)
Cl(2)	734(9)	424(7)	455(7)	34(6)	312(7)	95(6)
C(51)	700(30)	330(20)	490(30)	40(20)	300(30)	-40(20)
Cl(3)	503(8)	511(7)	487(7)	-128(6)	90(6)	-92(6)
Cl(4)	754(10)	829(10)	367(7)	-23(7)	156(7)	-216(8)
C(52)	470(30)	510(30)	310(30)	-100(20)	80(20)	-110(20)

Table 10. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 5 (CCDC 172948).

	x	y	z	U_{iso}
H(1A)	4971(18)	3384(15)	6477(15)	2(8)
H(1B)	5210(18)	3355(15)	7285(16)	2(8)
H(2A)	6910(20)	1963(17)	7315(16)	15(9)
H(2B)	7500(20)	2546(17)	7810(17)	17(9)
H(4)	3850(18)	3277(16)	7365(15)	0(8)
H(5)	2482(19)	3530(17)	7272(16)	12(9)
H(6)	1330(20)	2811(18)	6550(18)	26(10)
H(7)	1630(20)	1808(19)	5923(18)	24(10)
H(8)	3051(19)	1595(16)	5963(16)	9(9)
H(10)	5560(20)	959(17)	6395(17)	14(9)
H(11)	5720(20)	493(18)	5323(17)	23(10)
H(12)	4961(19)	1137(16)	4200(17)	15(9)
H(13)	4120(20)	2178(18)	4217(17)	20(10)
H(14)	3990(18)	2613(15)	5259(14)	0(8)
H(16)	7470(20)	2277(17)	6487(17)	13(9)
H(17)	7630(20)	2203(18)	5387(18)	20(10)
H(18)	6800(20)	2941(17)	4384(17)	19(9)
H(19)	5790(20)	3702(19)	4559(19)	27(11)
H(20)	5630(20)	3852(17)	5669(17)	19(10)
H(22)	8110(20)	3701(18)	7685(16)	18(9)
H(23)	8620(20)	4748(17)	8251(16)	9(9)
H(24)	7840(20)	5795(17)	8447(16)	15(9)
H(25)	6260(20)	5658(18)	7878(17)	25(10)
H(26)	5708(19)	4579(16)	7231(16)	6(8)
H(28)	7530(20)	3374(17)	8966(17)	20(10)
H(29)	7290(20)	4421(18)	9587(17)	15(10)
H(30)	6020(20)	4636(19)	9707(18)	27(11)
H(31)	4900(20)	3781(18)	9284(17)	18(10)
H(32)	5120(20)	2736(17)	8694(16)	11(9)
H(34)	6980(20)	1769(16)	9808(16)	11(9)
H(35)	8000(20)	1065(18)	10597(18)	16(11)
H(36)	8970(20)	391(18)	10341(18)	21(10)
H(37)	8960(20)	467(17)	9147(16)	11(9)
H(38)	7933(19)	1182(16)	8319(16)	9(9)
H(39)	6433(19)	-50(16)	8143(15)	6(8)
H(40)	7060(20)	-926(18)	9026(17)	21(10)
H(41)	6820(20)	-849(18)	10134(17)	23(10)
H(42)	5844(18)	109(15)	10248(15)	5(8)
H(45)	5610(20)	1519(17)	10286(17)	15(9)
H(46)	4650(20)	2446(19)	10357(19)	32(11)
H(47)	3440(20)	2612(18)	9364(17)	12(10)
H(48)	2990(20)	1971(17)	8258(17)	18(9)
H(49)	3977(18)	964(15)	8226(15)	4(8)
H(51A)	4878	8012	7755	73
H(51B)	5922	7868	8128	73
H(52A)	2350	230	7615	68
H(52B)	1789	1005	7358	68

Figure 3. Labeled drawing of 8 (CCDC 186799), with 50% ellipsoids. Hydrogen atoms have been omitted for clarity.

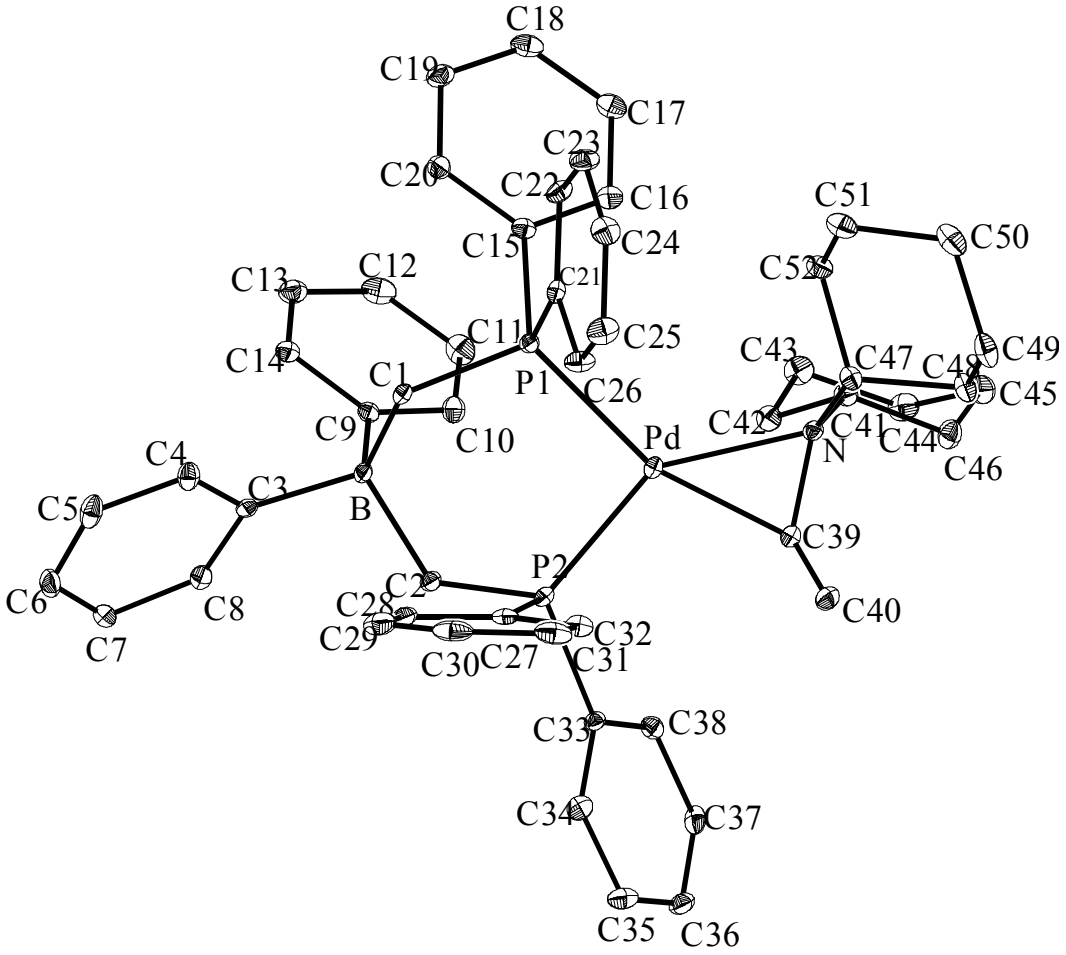

Table 11. Crystal Data and Structure Analysis Details for 8 (CCDC 186799).

Empirical formula	$C_{52}H_{60}BNP_2Pd$
Formula weight	878.16
Crystallization solvent	CH_2Cl_2
Crystal shape	tabular
Crystal color	colorless
Crystal size	0.10 x 0.14 x 0.15 mm

Data Collection

Preliminary photograph(s)	rotation	
Type of diffractometer	Bruker SMART 1000 ccd	
Wavelength	0.71073 Å MoKα	
Data collection temperature	98 K	
Theta range for 6911 reflections used in lattice determination	2.40 to 28.67°	
Unit cell dimensions	a = 12.9776(11) Å b = 19.4418(16) Å c = 18.1205(15) Å	α= 90° β= 107.570(1)° γ = 90°
Volume	4358.7(6) $Å^3$	
Z	4	
Crystal system	monoclinic	
Space group	*P* 2_1/c (# 14)	
Density (calculated)	1.338 g/cm^3	
F(000)	1840	
Theta range for data collection	1.6 to 28.5°	
Completeness to theta = 28.50°	94.1%	
Index ranges	$-17 \le h \le 17$, $-26 \le k \le 25$, $-23 \le l \le 24$	
Data collection scan type	ω scans at 7 fixed ϕ values	
Reflections collected	77250	
Independent reflections	10404 [R_{int}= 0.0721]	
Reflections > 2σ(I)	7523	
Average σ(I)/(net I)	0.0550	
Absorption coefficient	0.54 mm^{-1}	
Absorption correction	integration	
Max. and min. transmission	0.9604 and 0.9191	
Reflections monitored for decay	first 75 scans recollected at end of runs	
Decay of standards	0%	

Structure Solution and Refinement

Primary solution method	direct methods
Secondary solution method	difference map
Hydrogen placement	calculated
Refinement method	full-matrix least-squares on F^2
Data / restraints / parameters	10404 / 0 / 515
Treatment of hydrogen atoms	not refined, U_{iso} fixed at 120% U_{eq} of attached atom
Goodness-of-fit on F^2	1.75
Final R indices [$I>2\sigma(I)$, 7523 reflections]	R1 = 0.0497, wR2 = 0.0953
R indices (all data)	R1 = 0.0777, wR2 = 0.0987
Type of weighting scheme used	calculated weights
Weighting scheme used	$w=1/[\sigma^2(F_o^2)+(0.02P)^2]$ where $P=(F_o^2+2F_c^2)/3$
Max shift/error	0.027
Average shift/error	0.001
Largest diff. peak and hole	4.14 and -1.68 e·Å^{-3}

Programs Used

Cell refinement	Bruker SMART v5.606, Bruker SAINT v6.29
Data collection	Bruker SMART v5.054
Data reduction	Bruker SAINT v6.29
Structure solution	SHELX-97 (Sheldrick, 1997)
Structure refinement	SHELX-97 (Sheldrick, 1997)
Graphics	Diamond, Bruker SHELXTL v6.12

References

Bruker (1999) SMART (v5.054), SMART (v5.606), SAINT (v6.29) and SHELXTL (v6.12). Bruker AXS Inc., Madison, Wisconsin, USA.

Diamond 2.1. (2000) Crystal Impact GbR, Bonn, Germany.

Spek, A.L. (1990). *Acta Cryst.,* A**46**, C-34.

Sheldrick, G. M. (1997). SHELX-97. Program for Structures Refinement. Univ. of Gottingen, Federal Republic of Germany.

Special Refinement Details

A small clear tabular crystal was selected and mounted on a glass fiber with Paratone-N oil. Seven runs of data were collected with 30 second long, –0.3° wide ω–scans at six values of φ (0, 51, 103, 154, 206, and 309°) with the detector 5 cm (nominal) distant at a θ of -28°. The initial cell for data reduction was calculated from 999 centered reflections chosen from throughout the data frames. A total of 2 reflections was discarded in the triclinic least-squares with a reciprocal lattice vector tolerance of 0.005. For data processing with SAINT v6.29, all defaults were used, except: a fixed box size of 1.8 x 1.8 x 0.7 was used, periodic orientation matrix updating was disabled, the instrument error was set to zero, no Laue class integration restraints were used, the I/σ lower limit and threshold were set to 5 and 4 respectively, and for the post-integration global least squares refinement, no constraints were applied. No decay correction was needed. A face-indexed absorption correction resulted in marginal improvement.

No reflections were specifically omitted from the final processed dataset; 1207 reflections were rejected, with 0 space group-absence violations, 23 inconsistent equivalents, and no reflections suppressed. Refinement of F^2 was against all reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement.

Of the twenty largest peaks in the final difference map, four are greater than |1| e·Å^{-3}. Three of these four peaks are near the palladium atom, including the largest positive peak of 4.14 e·Å^{-3} at a distance of 0.91 Å. The largest negative peak of –1.68 e·Å^{-3} is 0.58 Å from Pd.

Table 12. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)for 8 (CCDC 186799). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Pd	3278.5(2)	8621.2(1)	7521.3(1)	18.0(1)
P(1)	1636.8(7)	9089.6(4)	7605.7(5)	16.7(2)
P(2)	3684.4(7)	9559.6(4)	6932.3(5)	17.3(2)
N	3772(2)	7569(1)	7870(2)	18(1)
B	2425(3)	10534(2)	7648(2)	18(1)
C(1)	1423(2)	9977(2)	7288(2)	17(1)
C(2)	3568(3)	10353(2)	7418(2)	20(1)
C(3)	2054(3)	11301(2)	7290(2)	19(1)
C(4)	1058(3)	11470(2)	6778(2)	26(1)
C(5)	810(3)	12129(2)	6481(2)	33(1)
C(6)	1576(3)	12639(2)	6671(2)	35(1)
C(7)	2575(3)	12496(2)	7192(2)	37(1)
C(8)	2795(3)	11837(2)	7498(2)	30(1)
C(9)	2670(2)	10538(2)	8589(2)	18(1)
C(10)	3396(3)	10096(2)	9089(2)	25(1)
C(11)	3518(3)	10060(2)	9875(2)	34(1)
C(12)	2911(3)	10488(2)	10189(2)	35(1)
C(13)	2196(3)	10939(2)	9723(2)	30(1)
C(14)	2077(3)	10966(2)	8935(2)	24(1)
C(15)	1233(3)	8999(2)	8483(2)	20(1)
C(16)	1687(3)	8473(2)	9001(2)	24(1)
C(17)	1336(3)	8348(2)	9641(2)	29(1)
C(18)	535(3)	8750(2)	9771(2)	29(1)
C(19)	91(3)	9283(2)	9277(2)	30(1)
C(20)	443(3)	9404(2)	8633(2)	26(1)
C(21)	559(2)	8612(2)	6900(2)	19(1)
C(22)	-342(3)	8337(2)	7026(2)	24(1)
C(23)	-1091(3)	7960(2)	6460(2)	31(1)
C(24)	-945(3)	7860(2)	5739(2)	31(1)
C(25)	-46(3)	8130(2)	5605(2)	34(1)
C(26)	703(3)	8495(2)	6173(2)	29(1)
C(27)	2841(3)	9642(2)	5928(2)	20(1)
C(28)	2231(3)	10228(2)	5643(2)	26(1)
C(29)	1538(3)	10236(2)	4897(2)	36(1)
C(30)	1440(3)	9679(2)	4425(2)	39(1)
C(31)	2053(3)	9098(2)	4684(2)	35(1)
C(32)	2754(3)	9079(2)	5437(2)	27(1)
C(33)	5055(2)	9560(2)	6838(2)	19(1)
C(34)	5288(3)	9752(2)	6169(2)	25(1)
C(35)	6329(3)	9720(2)	6125(2)	29(1)
C(36)	7154(3)	9496(2)	6748(2)	28(1)
C(37)	6958(3)	9326(2)	7428(2)	28(1)
C(38)	5900(3)	9350(2)	7472(2)	23(1)
C(39)	4428(3)	7922(2)	7478(2)	22(1)
C(40)	4484(3)	7687(2)	6698(2)	27(1)
C(41)	4288(3)	7446(2)	8720(2)	24(1)

C(42)	4665(3)	8108(2)	9166(2)	25(1)
C(43)	5065(3)	7974(2)	10036(2)	34(1)
C(44)	5984(3)	7448(2)	10226(2)	34(1)
C(45)	5656(3)	6792(2)	9764(2)	35(1)
C(46)	5221(3)	6931(2)	8898(2)	32(1)
C(47)	2952(3)	7075(2)	7415(2)	22(1)
C(48)	3371(3)	6347(2)	7328(2)	29(1)
C(49)	2504(3)	5968(2)	6712(2)	37(1)
C(50)	1447(3)	5929(2)	6931(2)	38(1)
C(51)	1071(3)	6633(2)	7102(2)	34(1)
C(52)	1958(3)	7026(2)	7698(2)	26(1)

Table 13. Bond lengths [Å] and angles [°] for 8 (CCDC 186799).

Pd-C(39)	2.037(3)	C(19)-C(20)	1.395(5)
Pd-N	2.180(3)	C(19)-H(19)	0.9500
Pd-P(2)	2.2549(9)	C(20)-H(20)	0.9500
Pd-P(1)	2.3641(9)	C(21)-C(22)	1.366(4)
P(1)-C(1)	1.813(3)	C(21)-C(26)	1.403(4)
P(1)-C(15)	1.827(3)	C(22)-C(23)	1.390(5)
P(1)-C(21)	1.837(3)	C(22)-H(22)	0.9500
P(2)-C(2)	1.806(3)	C(23)-C(24)	1.388(5)
P(2)-C(27)	1.825(3)	C(23)-H(23)	0.9500
P(2)-C(33)	1.838(3)	C(24)-C(25)	1.366(5)
N-C(39)	1.438(4)	C(24)-H(24)	0.9500
N-C(47)	1.484(4)	C(25)-C(26)	1.381(5)
N-C(41)	1.502(4)	C(25)-H(25)	0.9500
B-C(9)	1.637(5)	C(26)-H(26)	0.9500
B-C(3)	1.639(5)	C(27)-C(32)	1.394(5)
B-C(1)	1.666(5)	C(27)-C(28)	1.395(4)
B-C(2)	1.694(5)	C(28)-C(29)	1.380(5)
C(1)-H(1A)	0.9900	C(28)-H(28)	0.9500
C(1)-H(1B)	0.9900	C(29)-C(30)	1.362(6)
C(2)-H(2A)	0.9900	C(29)-H(29)	0.9500
C(2)-H(2B)	0.9900	C(30)-C(31)	1.379(6)
C(3)-C(4)	1.384(5)	C(30)-H(30)	0.9500
C(3)-C(8)	1.392(5)	C(31)-C(32)	1.393(5)
C(4)-C(5)	1.388(5)	C(31)-H(31)	0.9500
C(4)-H(4)	0.9500	C(32)-H(32)	0.9500
C(5)-C(6)	1.373(5)	C(33)-C(34)	1.386(4)
C(5)-H(5)	0.9500	C(33)-C(38)	1.388(4)
C(6)-C(7)	1.382(5)	C(34)-C(35)	1.379(5)
C(6)-H(6)	0.9500	C(34)-H(34)	0.9500
C(7)-C(8)	1.390(5)	C(35)-C(36)	1.371(5)
C(7)-H(7)	0.9500	C(35)-H(35)	0.9500
C(8)-H(8)	0.9500	C(36)-C(37)	1.371(5)
C(9)-C(10)	1.389(4)	C(36)-H(36)	0.9500
C(9)-C(14)	1.403(4)	C(37)-C(38)	1.401(5)
C(10)-C(11)	1.387(5)	C(37)-H(37)	0.9500
C(10)-H(10)	0.9500	C(38)-H(38)	0.9500
C(11)-C(12)	1.382(5)	C(39)-C(40)	1.508(4)
C(11)-H(11)	0.9500	C(39)-H(39)	1.0000
C(12)-C(13)	1.367(5)	C(40)-H(40A)	0.9800
C(12)-H(12)	0.9500	C(40)-H(40B)	0.9800
C(13)-C(14)	1.390(4)	C(40)-H(40C)	0.9800
C(13)-H(13)	0.9500	C(41)-C(42)	1.520(5)
C(14)-H(14)	0.9500	C(41)-C(46)	1.529(4)
C(15)-C(20)	1.383(4)	C(41)-H(41)	1.0000
C(15)-C(16)	1.393(4)	C(42)-C(43)	1.526(5)
C(16)-C(17)	1.390(5)	C(42)-H(42A)	0.9900
C(16)-H(16)	0.9500	C(42)-H(42B)	0.9900
C(17)-C(18)	1.377(5)	C(43)-C(44)	1.530(5)
C(17)-H(17)	0.9500	C(43)-H(43A)	0.9900
C(18)-C(19)	1.376(5)	C(43)-H(43B)	0.9900
C(18)-H(18)	0.9500	C(44)-C(45)	1.513(5)

C(44)-H(44A)	0.9900	C(1)-B-C(2)	114.0(3)
C(44)-H(44B)	0.9900	B-C(1)-P(1)	117.9(2)
C(45)-C(46)	1.523(5)	B-C(1)-H(1A)	107.8
C(45)-H(45A)	0.9900	P(1)-C(1)-H(1A)	107.8
C(45)-H(45B)	0.9900	B-C(1)-H(1B)	107.8
C(46)-H(46A)	0.9900	P(1)-C(1)-H(1B)	107.8
C(46)-H(46B)	0.9900	H(1A)-C(1)-H(1B)	107.2
C(47)-C(52)	1.528(5)	B-C(2)-P(2)	120.4(2)
C(47)-C(48)	1.542(4)	B-C(2)-H(2A)	107.2
C(47)-H(47)	1.0000	P(2)-C(2)-H(2A)	107.2
C(48)-C(49)	1.515(5)	B-C(2)-H(2B)	107.2
C(48)-H(48A)	0.9900	P(2)-C(2)-H(2B)	107.2
C(48)-H(48B)	0.9900	H(2A)-C(2)-H(2B)	106.9
C(49)-C(50)	1.541(5)	C(4)-C(3)-C(8)	115.6(3)
C(49)-H(49A)	0.9900	C(4)-C(3)-B	125.9(3)
C(49)-H(49B)	0.9900	C(8)-C(3)-B	118.5(3)
C(50)-C(51)	1.514(5)	C(3)-C(4)-C(5)	122.7(3)
C(50)-H(50A)	0.9900	C(3)-C(4)-H(4)	118.7
C(50)-H(50B)	0.9900	C(5)-C(4)-H(4)	118.7
C(51)-C(52)	1.525(5)	C(6)-C(5)-C(4)	120.2(4)
C(51)-H(51A)	0.9900	C(6)-C(5)-H(5)	119.9
C(51)-H(51B)	0.9900	C(4)-C(5)-H(5)	119.9
C(52)-H(52A)	0.9900	C(5)-C(6)-C(7)	119.0(3)
C(52)-H(52B)	0.9900	C(5)-C(6)-H(6)	120.5
		C(7)-C(6)-H(6)	120.5
C(39)-Pd-N	39.70(11)	C(6)-C(7)-C(8)	119.7(4)
C(39)-Pd-P(2)	104.04(9)	C(6)-C(7)-H(7)	120.2
N-Pd-P(2)	143.75(7)	C(8)-C(7)-H(7)	120.2
C(39)-Pd-P(1)	160.81(9)	C(7)-C(8)-C(3)	122.7(4)
N-Pd-P(1)	122.31(7)	C(7)-C(8)-H(8)	118.7
P(2)-Pd-P(1)	93.60(3)	C(3)-C(8)-H(8)	118.7
C(1)-P(1)-C(15)	108.13(15)	C(10)-C(9)-C(14)	115.5(3)
C(1)-P(1)-C(21)	104.40(14)	C(10)-C(9)-B	123.8(3)
C(15)-P(1)-C(21)	101.48(14)	C(14)-C(9)-B	120.5(3)
C(1)-P(1)-Pd	113.62(10)	C(11)-C(10)-C(9)	123.1(3)
C(15)-P(1)-Pd	121.34(11)	C(11)-C(10)-H(10)	118.4
C(21)-P(1)-Pd	105.77(10)	C(9)-C(10)-H(10)	118.4
C(2)-P(2)-C(27)	107.36(15)	C(12)-C(11)-C(10)	119.3(3)
C(2)-P(2)-C(33)	105.75(14)	C(12)-C(11)-H(11)	120.4
C(27)-P(2)-C(33)	102.31(14)	C(10)-C(11)-H(11)	120.4
C(2)-P(2)-Pd	113.23(11)	C(13)-C(12)-C(11)	119.8(3)
C(27)-P(2)-Pd	112.79(11)	C(13)-C(12)-H(12)	120.1
C(33)-P(2)-Pd	114.51(11)	C(11)-C(12)-H(12)	120.1
C(39)-N-C(47)	117.9(3)	C(12)-C(13)-C(14)	120.2(3)
C(39)-N-C(41)	115.8(3)	C(12)-C(13)-H(13)	119.9
C(47)-N-C(41)	119.3(2)	C(14)-C(13)-H(13)	119.9
C(39)-N-Pd	64.78(16)	C(13)-C(14)-C(9)	122.1(3)
C(47)-N-Pd	110.33(18)	C(13)-C(14)-H(14)	119.0
C(41)-N-Pd	116.58(19)	C(9)-C(14)-H(14)	119.0
C(9)-B-C(3)	110.5(3)	C(20)-C(15)-C(16)	118.3(3)
C(9)-B-C(1)	107.0(3)	C(20)-C(15)-P(1)	122.7(3)
C(3)-B-C(1)	109.6(3)	C(16)-C(15)-P(1)	118.9(2)
C(9)-B-C(2)	110.3(3)	C(17)-C(16)-C(15)	120.8(3)
C(3)-B-C(2)	105.4(3)	C(17)-C(16)-H(16)	119.6

C(15)-C(16)-H(16)	119.6	C(33)-C(34)-H(34)	119.5
C(18)-C(17)-C(16)	119.8(3)	C(36)-C(35)-C(34)	120.2(3)
C(18)-C(17)-H(17)	120.1	C(36)-C(35)-H(35)	119.9
C(16)-C(17)-H(17)	120.1	C(34)-C(35)-H(35)	119.9
C(19)-C(18)-C(17)	120.4(3)	C(35)-C(36)-C(37)	120.4(3)
C(19)-C(18)-H(18)	119.8	C(35)-C(36)-H(36)	119.8
C(17)-C(18)-H(18)	119.8	C(37)-C(36)-H(36)	119.8
C(18)-C(19)-C(20)	119.5(3)	C(36)-C(37)-C(38)	119.6(3)
C(18)-C(19)-H(19)	120.2	C(36)-C(37)-H(37)	120.2
C(20)-C(19)-H(19)	120.2	C(38)-C(37)-H(37)	120.2
C(15)-C(20)-C(19)	121.1(3)	C(33)-C(38)-C(37)	120.3(3)
C(15)-C(20)-H(20)	119.4	C(33)-C(38)-H(38)	119.8
C(19)-C(20)-H(20)	119.4	C(37)-C(38)-H(38)	119.8
C(22)-C(21)-C(26)	117.1(3)	N-C(39)-C(40)	121.4(3)
C(22)-C(21)-P(1)	126.3(2)	N-C(39)-Pd	75.51(17)
C(26)-C(21)-P(1)	116.5(2)	C(40)-C(39)-Pd	118.8(2)
C(21)-C(22)-C(23)	121.7(3)	N-C(39)-H(39)	112.1
C(21)-C(22)-H(22)	119.1	C(40)-C(39)-H(39)	112.1
C(23)-C(22)-H(22)	119.1	Pd-C(39)-H(39)	112.1
C(24)-C(23)-C(22)	120.3(3)	C(39)-C(40)-H(40A)	109.5
C(24)-C(23)-H(23)	119.9	C(39)-C(40)-H(40B)	109.5
C(22)-C(23)-H(23)	119.9	H(40A)-C(40)-H(40B)	109.5
C(25)-C(24)-C(23)	118.7(3)	C(39)-C(40)-H(40C)	109.5
C(25)-C(24)-H(24)	120.6	H(40A)-C(40)-H(40C)	109.5
C(23)-C(24)-H(24)	120.6	H(40B)-C(40)-H(40C)	109.5
C(24)-C(25)-C(26)	120.7(3)	N-C(41)-C(42)	112.5(3)
C(24)-C(25)-H(25)	119.7	N-C(41)-C(46)	113.6(3)
C(26)-C(25)-H(25)	119.7	C(42)-C(41)-C(46)	109.6(3)
C(25)-C(26)-C(21)	121.4(3)	N-C(41)-H(41)	106.9
C(25)-C(26)-H(26)	119.3	C(42)-C(41)-H(41)	106.9
C(21)-C(26)-H(26)	119.3	C(46)-C(41)-H(41)	106.9
C(32)-C(27)-C(28)	118.6(3)	C(41)-C(42)-C(43)	111.1(3)
C(32)-C(27)-P(2)	118.5(3)	C(41)-C(42)-H(42A)	109.4
C(28)-C(27)-P(2)	122.9(3)	C(43)-C(42)-H(42A)	109.4
C(29)-C(28)-C(27)	120.0(4)	C(41)-C(42)-H(42B)	109.4
C(29)-C(28)-H(28)	120.0	C(43)-C(42)-H(42B)	109.4
C(27)-C(28)-H(28)	120.0	H(42A)-C(42)-H(42B)	108.0
C(30)-C(29)-C(28)	121.1(4)	C(42)-C(43)-C(44)	110.2(3)
C(30)-C(29)-H(29)	119.4	C(42)-C(43)-H(43A)	109.6
C(28)-C(29)-H(29)	119.4	C(44)-C(43)-H(43A)	109.6
C(29)-C(30)-C(31)	120.2(4)	C(42)-C(43)-H(43B)	109.6
C(29)-C(30)-H(30)	119.9	C(44)-C(43)-H(43B)	109.6
C(31)-C(30)-H(30)	119.9	H(43A)-C(43)-H(43B)	108.1
C(30)-C(31)-C(32)	119.6(4)	C(45)-C(44)-C(43)	111.7(3)
C(30)-C(31)-H(31)	120.2	C(45)-C(44)-H(44A)	109.3
C(32)-C(31)-H(31)	120.2	C(43)-C(44)-H(44A)	109.3
C(31)-C(32)-C(27)	120.5(4)	C(45)-C(44)-H(44B)	109.3
C(31)-C(32)-H(32)	119.7	C(43)-C(44)-H(44B)	109.3
C(27)-C(32)-H(32)	119.7	H(44A)-C(44)-H(44B)	107.9
C(34)-C(33)-C(38)	118.5(3)	C(44)-C(45)-C(46)	112.1(3)
C(34)-C(33)-P(2)	123.6(3)	C(44)-C(45)-H(45A)	109.2
C(38)-C(33)-P(2)	117.9(2)	C(46)-C(45)-H(45A)	109.2
C(35)-C(34)-C(33)	121.0(3)	C(44)-C(45)-H(45B)	109.2
C(35)-C(34)-H(34)	119.5	C(46)-C(45)-H(45B)	109.2

H(45A)-C(45)-H(45B)	107.9	C(48)-C(49)-H(49B)	109.5
C(45)-C(46)-C(41)	110.9(3)	C(50)-C(49)-H(49B)	109.5
C(45)-C(46)-H(46A)	109.5	H(49A)-C(49)-H(49B)	108.1
C(41)-C(46)-H(46A)	109.5	C(51)-C(50)-C(49)	112.0(3)
C(45)-C(46)-H(46B)	109.5	C(51)-C(50)-H(50A)	109.2
C(41)-C(46)-H(46B)	109.5	C(49)-C(50)-H(50A)	109.2
H(46A)-C(46)-H(46B)	108.0	C(51)-C(50)-H(50B)	109.2
N-C(47)-C(52)	113.0(3)	C(49)-C(50)-H(50B)	109.2
N-C(47)-C(48)	115.8(3)	H(50A)-C(50)-H(50B)	107.9
C(52)-C(47)-C(48)	109.7(3)	C(50)-C(51)-C(52)	112.1(3)
N-C(47)-H(47)	105.9	C(50)-C(51)-H(51A)	109.2
C(52)-C(47)-H(47)	105.9	C(52)-C(51)-H(51A)	109.2
C(48)-C(47)-H(47)	105.9	C(50)-C(51)-H(51B)	109.2
C(49)-C(48)-C(47)	108.3(3)	C(52)-C(51)-H(51B)	109.2
C(49)-C(48)-H(48A)	110.0	H(51A)-C(51)-H(51B)	107.9
C(47)-C(48)-H(48A)	110.0	C(51)-C(52)-C(47)	109.1(3)
C(49)-C(48)-H(48B)	110.0	C(51)-C(52)-H(52A)	109.9
C(47)-C(48)-H(48B)	110.0	C(47)-C(52)-H(52A)	109.9
H(48A)-C(48)-H(48B)	108.4	C(51)-C(52)-H(52B)	109.9
C(48)-C(49)-C(50)	110.8(3)	C(47)-C(52)-H(52B)	109.9
C(48)-C(49)-H(49A)	109.5	H(52A)-C(52)-H(52B)	108.3
C(50)-C(49)-H(49A)	109.5		

Table 14. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for 8 (CCDC 186799). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd	167(1)	178(1)	191(1)	-3(1)	50(1)	36(1)
P(1)	159(4)	167(4)	179(5)	-7(3)	57(3)	-1(3)
P(2)	134(4)	201(4)	189(4)	-19(3)	57(4)	17(3)
N	145(14)	199(14)	188(15)	18(11)	48(12)	21(11)
B	140(18)	160(17)	230(20)	-6(15)	59(16)	17(14)
C(1)	147(16)	193(16)	170(17)	-2(13)	46(13)	42(12)
C(2)	175(17)	190(16)	246(19)	-35(14)	88(15)	-13(13)
C(3)	240(17)	200(17)	191(17)	-15(14)	140(14)	9(14)
C(4)	320(20)	224(18)	213(18)	-38(15)	63(15)	44(15)
C(5)	410(20)	340(20)	200(20)	9(16)	54(17)	171(18)
C(6)	560(30)	239(19)	340(20)	115(17)	270(20)	149(19)
C(7)	370(20)	199(18)	630(30)	48(18)	300(20)	4(16)
C(8)	248(19)	212(18)	460(20)	6(17)	155(18)	21(15)
C(9)	163(16)	163(16)	222(18)	-22(13)	47(14)	-42(13)
C(10)	262(19)	232(18)	240(20)	-17(15)	48(16)	13(15)
C(11)	300(20)	380(20)	270(20)	59(17)	-12(17)	1(17)
C(12)	390(20)	470(20)	172(19)	2(17)	73(17)	-84(19)
C(13)	320(20)	320(20)	290(20)	-39(17)	159(17)	-43(17)
C(14)	248(19)	245(18)	245(19)	35(15)	107(15)	5(15)
C(15)	214(18)	210(17)	182(17)	-35(14)	61(14)	-57(14)
C(16)	204(18)	300(20)	208(18)	-8(14)	43(15)	-28(14)
C(17)	300(20)	340(20)	211(19)	24(15)	51(16)	-70(16)
C(18)	340(20)	380(20)	179(18)	-55(16)	108(16)	-129(17)
C(19)	320(20)	310(20)	330(20)	-95(17)	192(18)	-44(16)
C(20)	310(20)	220(18)	260(20)	-6(15)	130(16)	-3(15)
C(21)	165(16)	161(15)	210(17)	-26(14)	24(13)	32(14)
C(22)	198(18)	320(19)	229(19)	-77(15)	87(15)	-10(15)
C(23)	204(19)	370(20)	370(20)	-48(17)	114(17)	-99(16)
C(24)	250(20)	320(20)	300(20)	-66(16)	3(17)	-89(16)
C(25)	350(20)	430(20)	240(20)	-96(17)	102(17)	-45(18)
C(26)	230(19)	380(20)	265(19)	-45(16)	106(16)	-89(16)
C(27)	159(17)	293(18)	175(17)	23(14)	81(14)	-42(14)
C(28)	192(18)	350(20)	280(20)	51(16)	123(16)	33(15)
C(29)	240(20)	550(30)	310(20)	170(20)	112(18)	63(18)
C(30)	178(19)	710(30)	250(20)	140(20)	21(16)	-107(19)
C(31)	350(20)	510(30)	220(20)	-72(18)	120(18)	-210(20)
C(32)	264(19)	310(20)	240(19)	13(16)	102(16)	-53(16)
C(33)	149(16)	185(16)	244(18)	-37(14)	81(14)	-21(13)
C(34)	191(18)	309(19)	227(19)	-28(15)	29(15)	-14(15)
C(35)	270(20)	380(20)	270(20)	-63(16)	141(17)	-83(16)
C(36)	206(19)	274(19)	410(20)	-71(17)	160(17)	-30(15)
C(37)	125(18)	210(17)	410(20)	-9(16)	-35(17)	10(13)
C(38)	241(19)	196(17)	248(19)	-10(14)	71(16)	-30(14)
C(39)	209(18)	202(17)	290(20)	25(14)	107(15)	38(14)
C(40)	290(20)	288(19)	290(20)	16(16)	151(17)	53(16)

C(41)	225(18)	273(18)	203(18)	70(15)	60(15)	30(15)
C(42)	218(19)	280(19)	226(19)	15(15)	22(15)	5(15)
C(43)	330(20)	400(20)	260(20)	14(17)	64(17)	17(17)
C(44)	240(20)	470(20)	260(20)	71(18)	14(17)	41(17)
C(45)	310(20)	390(20)	340(20)	159(18)	64(18)	84(18)
C(46)	290(20)	320(20)	320(20)	45(17)	55(17)	97(16)
C(47)	233(18)	201(17)	219(19)	14(14)	44(15)	-7(14)
C(48)	340(20)	184(16)	330(20)	32(16)	67(16)	8(16)
C(49)	520(30)	192(19)	360(20)	-3(16)	80(20)	6(17)
C(50)	410(20)	290(20)	380(20)	65(18)	23(19)	-82(18)
C(51)	320(20)	350(20)	340(20)	34(17)	91(18)	-97(17)
C(52)	250(20)	281(19)	250(20)	48(15)	78(16)	-32(15)

Table 15. Hydrogen coordinates (x 10^3) and isotropic displacement parameters (Å^{2}x 10^3) for 8 (CCDC 186799).

	x	y	z	U_{iso}
H(1A)	125	998	672	20
H(1B)	78	1015	741	20
H(2A)	417	1037	790	24
H(2B)	368	1073	709	24
H(4)	52	1112	662	31
H(5)	11	1223	614	39
H(6)	142	1308	645	42
H(7)	311	1284	734	44
H(8)	348	1175	786	35
H(10)	383	981	888	30
H(11)	401	974	1019	41
H(12)	299	1047	1073	42
H(13)	178	1124	994	36
H(14)	158	1128	862	29
H(16)	224	820	892	29
H(17)	165	798	999	35
H(18)	29	866	1020	35
H(19)	-45	956	937	36
H(20)	13	977	829	31
H(22)	-46	840	751	29
H(23)	-170	777	657	37
H(24)	-146	761	535	37
H(25)	6	807	511	40
H(26)	133	867	607	34
H(28)	229	1062	596	32
H(29)	112	1064	471	43
H(30)	95	969	392	47
H(31)	200	871	435	42
H(32)	318	868	562	32
H(34)	472	991	574	30
H(35)	648	985	566	35
H(36)	787	946	671	34
H(37)	754	919	787	33
H(38)	576	922	794	27
H(39)	516	804	784	27
H(40A)	487	725	675	33
H(40B)	487	803	649	33
H(40C)	375	763	635	33
H(41)	372	724	892	28
H(42A)	406	844	905	30
H(42B)	526	831	900	30
H(43A)	446	780	1021	40
H(43B)	533	841	1031	40
H(44A)	662	765	1011	40
H(44B)	620	734	1078	40
H(45A)	510	655	994	42
H(45B)	629	648	987	42

H(46A)	497	649	862	38
H(46B)	581	711	871	38
H(47)	269	727	688	27
H(48A)	404	638	718	35
H(48B)	354	610	783	35
H(49A)	276	550	665	44
H(49B)	237	621	621	44
H(50A)	88	572	650	46
H(50B)	156	563	739	46
H(51A)	44	658	730	41
H(51B)	84	690	662	41
H(52A)	215	678	820	31
H(52B)	170	749	777	31

Figure 4. Labeled drawing of 9 (CCDC 186800), with 50% ellipsoids. Hydrogen atoms have been omitted for clarity.

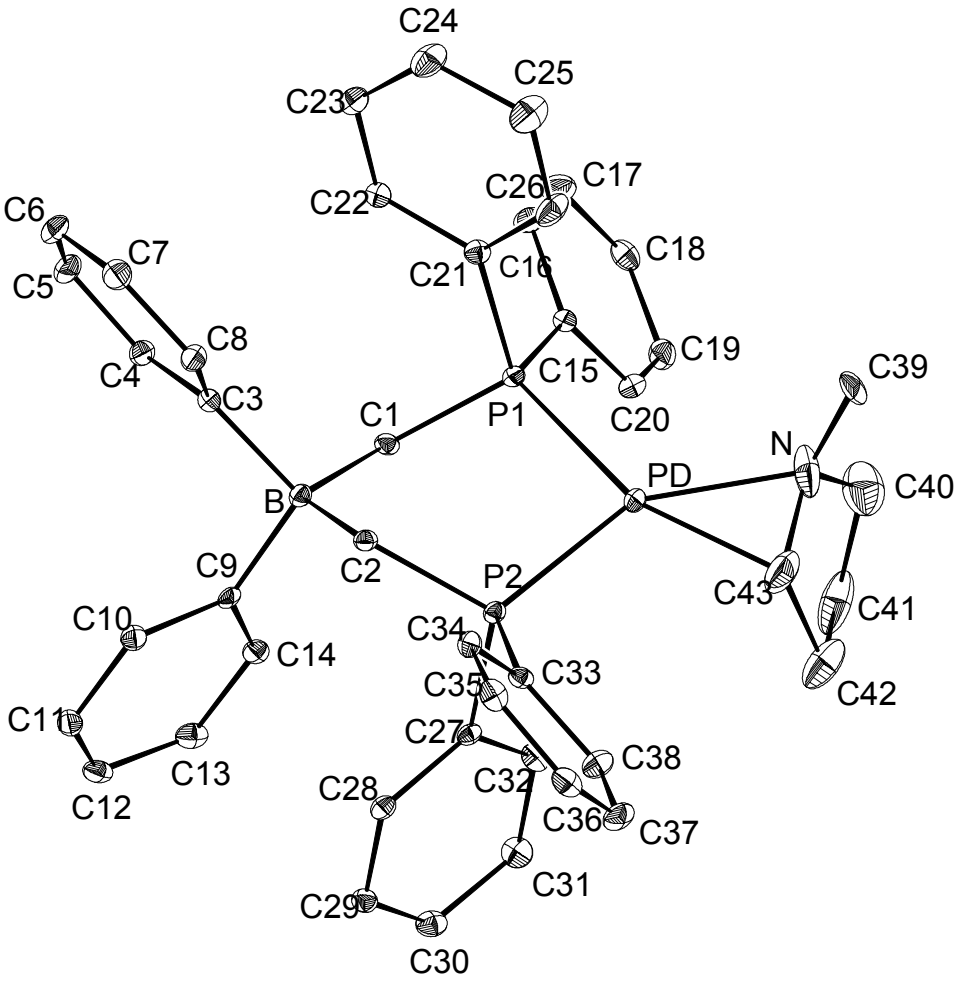

Table 16. Crystal Data and Structure Analysis Details for 9 (CCDC 186800).

Empirical formula	$C_{43}H_{44}BNP_2Pd$
Formula weight	753.94
Crystallization solvent	tetrahydrofuran
Crystal shape	blade
Crystal color	colorless
Crystal size	0.11 x 0.12 x 0.30 mm

Data Collection

Preliminary photograph(s)	rotation	
Type of diffractometer	Bruker SMART 1000 ccd	
Wavelength	0.71073 Å MoKα	
Data collection temperature	98 K	
Theta range for 8067 reflections used in lattice determination	2.22 to 28.41°	
Unit cell dimensions	a = 11.6163(10) Å b = 29.268(2) Å c = 11.5305(9) Å	α= 90° β= 113.573 (1)° γ = 90°
Volume	3593.1(5) $Å^3$	
Z	4	
Crystal system	monoclinic	
Space group	$P\ 2_1/c$ (# 14)	
Density (calculated)	1.394 g/cm^3	
F(000)	1560	
Theta range for data collection	1.4 to 28.5°	
Completeness to theta = 28.52°	95.0%	
Index ranges	$-15 \le h \le 15, -38 \le k \le 38, -15 \le l \le 15$	
Data collection scan type	ω scans at 7 fixed ϕ values	
Reflections collected	83659	
Independent reflections	8682 [R_{int}= 0.0784]	
Reflections > 2σ(I)	6031	
Average σ(I)/(net I)	0.060	
Absorption coefficient	0.64 mm^{-1}	
Absorption correction	none	
Reflections monitored for decay	first 620 scans recollected at end of runs	
Decay of standards	0%	

Structure Solution and Refinement

Primary solution method	direct methods
Secondary solution method	difference map
Hydrogen placement	calculated
Refinement method	full-matrix least-squares on F^2
Data / restraints / parameters	8682 / 0 / 440
Treatment of hydrogen atoms	not refined, U_{iso} fixed at 120% U_{eq} of attached atom
Goodness-of-fit on F^2	1.61
Final R indices [$I>2\sigma(I)$, 6031 reflections]	R1 = 0.0485, wR2 = 0.0870
R indices (all data)	R1 = 0.0806, wR2 = 0.0900
Type of weighting scheme used	calculated weights
Weighting scheme used	calc $w=1/[\sigma^2(F_o^2)+(0.02P)^2]$ where $P=(F_o^2+2F_c^2)/3$
Max shift/error	0.002
Average shift/error	0.000
Largest diff. peak and hole	1.49 and -1.48 e·Å^{-3}

Programs Used

Cell refinement	Bruker SMART v5.606, Bruker SAINT v6.29
Data collection	Bruker SMART v5.054
Data reduction	Bruker SAINT v6.29
Structure solution	SHELX-97 (Sheldrick, 1997)
Structure refinement	SHELX-97 (Sheldrick, 1997)
Graphics	Diamond, Bruker SHELXTL v6.12

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Special Refinement Details

A colorless blade-shaped crystal was selected and mounted on a glass fiber with Paratone-N oil. Seven runs of data were collected with 30 second long, –0.3° wide ω–scans at seven values of φ (0, 51, 103, 154, 206, 257, and 309°) with the detector 5 cm (nominal) distant at a θ of -28°. The initial cell for data reduction was calculated from 999 centered reflections chosen from throughout the data frames. A total of 2 reflections was discarded in the triclinic least-squares with a reciprocal lattice vector tolerance of 0.005. For data processing with SAINT v6.29, all defaults were used, except: a fixed box size of 1.8 x 1.8 x 0.7 was used, periodic orientation matrix updating was disabled, the instrument error was set to zero, no Laue class integration restraints were used, the I/σ lower limit and threshold were set to 5 and 4 respectively, and for the post-integration global least squares refinement, no constraints were applied. No decay correction was needed.

No reflections were specifically omitted from the final processed dataset; 1047 reflections were rejected, with 8 space group-absence violations, 7 inconsistent equivalents, and no reflections suppressed. Refinement of F^2 was against all reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement.

The *N*-methylpyrrolidene fragment is coordinated to the Pd metal by C(43) and N. The difference map indicated this fragment was disordered. The structure was modeled as a superposition of two molecules in which C(43) and N interchange positions; the other ring carbon atoms in the two orientations overlap, and the *N*-methyl carbon occupies two different positions. (The minor position of the methyl carbon was refined isotropically.) The relative populations are 0.81 and 0.19. The two models have different geometries; there is a difference of ~15 degrees in both the P-Pd-N and P-Pd-C angles. In at least one similar system, the ligand is disordered about the molecular 2-fold axis bisecting the P-Pd-P angle, resulting in both models having the same geometry.

Of the twenty largest peaks in the final difference map, three are greater than |1| e·Å^{-3}. All three peaks are near the palladium atom (within a distance of 0.88 Å), including the largest positive peak of 1.49 e·Å^{-3} and the largest negative peak of –1.48 e·Å^{-3}.

Table 17. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 9 (CCDC 186800). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq} or U_{iso}
Pd	-343.9(2)	1290.8(1)	1749.2(2)	24.2(1)
P(1)	-105.3(8)	1626.2(3)	3669.9(7)	19.6(2)
P(2)	1606.2(7)	957.5(3)	2577.5(7)	18.4(2)
N[a]	-1964(3)	1408.3(15)	96(3)	63.4(12)
C(43')[b]	-1964(3)	1408.3(15)	96(3)	63.4(12)
B	2455(3)	1248.1(12)	5266(3)	19.5(7)
C(1)	948(3)	1298.5(11)	5007(3)	20.0(7)
C(2)	2694(3)	1254.2(10)	3934(3)	18.9(6)
C(3)	3265(3)	1665.7(10)	6192(3)	20.7(7)
C(4)	3231(3)	1715.7(11)	7391(3)	26.6(8)
C(5)	3831(3)	2068.7(12)	8198(3)	36.7(9)
C(6)	4518(3)	2386.3(12)	7856(3)	37.7(9)
C(7)	4598(3)	2345.8(11)	6707(3)	33.6(8)
C(8)	3969(3)	1993.3(11)	5886(3)	26.2(7)
C(9)	3005(3)	768.2(10)	6040(3)	18.3(7)
C(10)	4305(3)	695.9(11)	6630(3)	25.4(8)
C(11)	4825(3)	302.9(12)	7301(3)	29.1(8)
C(12)	4068(3)	-37.7(11)	7435(3)	29.8(8)
C(13)	2793(3)	16.9(11)	6857(3)	28.2(8)
C(14)	2281(3)	408.7(10)	6171(3)	22.5(7)
C(15)	-1543(3)	1633.2(10)	3969(3)	20.3(7)
C(16)	-1752(3)	1946.9(12)	4769(3)	31.9(8)
C(17)	-2772(3)	1900.7(12)	5104(3)	36.7(9)
C(18)	-3604(3)	1548.6(12)	4622(3)	31.3(8)
C(19)	-3418(3)	1238.8(12)	3818(3)	31.9(8)
C(20)	-2402(3)	1282.4(11)	3499(3)	27.6(7)
C(21)	397(3)	2219.8(10)	3907(3)	22.4(7)
C(22)	1043(3)	2409.1(11)	5103(3)	25.5(7)
C(23)	1402(3)	2861.9(11)	5232(3)	31.7(8)
C(24)	1107(4)	3136.7(12)	4182(4)	41.2(10)
C(25)	435(4)	2958.4(13)	2987(3)	47.9(11)
C(26)	99(4)	2503.1(12)	2852(3)	38.2(9)
C(27)	1608(3)	358.7(10)	3007(3)	18.0(7)
C(28)	2737(3)	118.9(10)	3568(3)	20.9(7)
C(29)	2743(3)	-335.5(11)	3881(3)	24.4(7)
C(30)	1631(3)	-562.2(11)	3632(3)	28.2(8)
C(31)	508(3)	-329.4(12)	3065(3)	31.0(8)
C(32)	501(3)	129.9(11)	2769(3)	25.4(7)
C(33)	2379(3)	920.1(10)	1472(3)	19.2(7)
C(34)	3369(3)	1196.9(10)	1559(3)	23.5(7)
C(35)	3912(3)	1160.8(11)	688(3)	29.5(8)
C(36)	3498(3)	837.4(12)	-256(3)	29.8(8)
C(37)	2517(3)	558.3(12)	-349(3)	32.3(9)
C(38)	1944(3)	601.8(11)	489(3)	30.0(8)
C(39)[a]	-2124(5)	1843.5(15)	-569(5)	52.0(17)
C(39')[b]	-1361(17)	1377(6)	-1196(17)	32(6)[c]

C(40)	-3072(5)	1182.5(19)	27(5)	81.5(16)
C(41)	-2759(5)	691.8(19)	238(4)	79.7(18)
C(42)	-1796(5)	631.0(17)	-348(4)	67.0(14)
C(43)[a]	1204(5)	1091.8(16)	-103(4)	61.3(13)
N'[b]	1204(5)	1091.8(16)	-103(4)	61.3(13)

[a] Population = 0.815(7)
[b] Population = 0.185(7)
[c] U_{iso}

Table 18. Bond lengths [Å] and angles [°] for 9 (CCDC 186800).

Pd-C(43)	2.048(4)	C(19)-C(20)	1.375(4)
Pd-N	2.104(3)	C(19)-H(19)	0.9500
Pd-P(2)	2.2945(8)	C(20)-H(20)	0.9500
Pd-P(1)	2.3354(8)	C(21)-C(22)	1.394(4)
P(1)-C(1)	1.812(3)	C(21)-C(26)	1.396(4)
P(1)-C(21)	1.818(3)	C(22)-C(23)	1.380(4)
P(1)-C(15)	1.835(3)	C(22)-H(22)	0.9500
P(2)-C(2)	1.793(3)	C(23)-C(24)	1.377(5)
P(2)-C(27)	1.821(3)	C(23)-H(23)	0.9500
P(2)-C(33)	1.832(3)	C(24)-C(25)	1.386(5)
N-C(43)	1.361(5)	C(24)-H(24)	0.9500
N-C(40)	1.420(6)	C(25)-C(26)	1.379(5)
N-C(39)	1.460(5)	C(25)-H(25)	0.9500
B-C(3)	1.648(4)	C(26)-H(26)	0.9500
B-C(9)	1.650(5)	C(27)-C(32)	1.376(4)
B-C(1)	1.661(4)	C(27)-C(28)	1.398(4)
B-C(2)	1.666(4)	C(28)-C(29)	1.377(4)
C(1)-H(1A)	0.9900	C(28)-H(28)	0.9500
C(1)-H(1B)	0.9900	C(29)-C(30)	1.376(4)
C(2)-H(2A)	0.9900	C(29)-H(29)	0.9500
C(2)-H(2B)	0.9900	C(30)-C(31)	1.383(4)
C(3)-C(8)	1.394(4)	C(30)-H(30)	0.9500
C(3)-C(4)	1.407(4)	C(31)-C(32)	1.386(4)
C(4)-C(5)	1.378(4)	C(31)-H(31)	0.9500
C(4)-H(4)	0.9500	C(32)-H(32)	0.9500
C(5)-C(6)	1.381(5)	C(33)-C(34)	1.378(4)
C(5)-H(5)	0.9500	C(33)-C(38)	1.396(4)
C(6)-C(7)	1.370(5)	C(34)-C(35)	1.388(4)
C(6)-H(6)	0.9500	C(34)-H(34)	0.9500
C(7)-C(8)	1.394(4)	C(35)-C(36)	1.375(4)
C(7)-H(7)	0.9500	C(35)-H(35)	0.9500
C(8)-H(8)	0.9500	C(36)-C(37)	1.370(5)
C(9)-C(14)	1.393(4)	C(36)-H(36)	0.9500
C(9)-C(10)	1.402(4)	C(37)-C(38)	1.382(4)
C(10)-C(11)	1.383(4)	C(37)-H(37)	0.9500
C(10)-H(10)	0.9500	C(38)-H(38)	0.9500
C(11)-C(12)	1.378(5)	C(39)-H(39A)	0.9800
C(11)-H(11)	0.9500	C(39)-H(39B)	0.9800
C(12)-C(13)	1.370(5)	C(39)-H(39C)	0.9800
C(12)-H(12)	0.9500	C(39')-H(39D)	0.9800
C(13)-C(14)	1.385(4)	C(39')-H(39E)	0.9800
C(13)-H(13)	0.9500	C(39')-H(39F)	0.9800
C(14)-H(14)	0.9500	C(40)-C(41)	1.478(6)
C(15)-C(20)	1.383(4)	C(40)-H(40A)	0.9900
C(15)-C(16)	1.390(4)	C(40)-H(40B)	0.9900
C(16)-C(17)	1.392(5)	C(41)-C(42)	1.530(6)
C(16)-H(16)	0.9500	C(41)-H(41A)	0.9900
C(17)-C(18)	1.369(5)	C(41)-H(41B)	0.9900
C(17)-H(17)	0.9500	C(42)-C(43)	1.488(6)
C(18)-C(19)	1.374(5)	C(42)-H(42A)	0.9900
C(18)-H(18)	0.9500	C(42)-H(42B)	0.9900

C(43)-H(43)	1.0000	C(5)-C(6)-H(6)	120.4
C(43)-Pd-N	38.25(16)	C(6)-C(7)-C(8)	120.1(3)
C(43)-Pd-P(2)	107.58(15)	C(6)-C(7)-H(7)	120.0
N-Pd-P(2)	145.42(13)	C(8)-C(7)-H(7)	120.0
C(43)-Pd-P(1)	158.75(15)	C(3)-C(8)-C(7)	122.6(3)
N-Pd-P(1)	120.96(13)	C(3)-C(8)-H(8)	118.7
P(2)-Pd-P(1)	93.49(3)	C(7)-C(8)-H(8)	118.7
C(1)-P(1)-C(21)	108.11(14)	C(14)-C(9)-C(10)	114.4(3)
C(1)-P(1)-C(15)	101.61(13)	C(14)-C(9)-B	125.6(3)
C(21)-P(1)-C(15)	102.87(14)	C(10)-C(9)-B	120.0(3)
C(1)-P(1)-Pd	111.59(10)	C(11)-C(10)-C(9)	122.8(3)
C(21)-P(1)-Pd	116.95(10)	C(11)-C(10)-H(10)	118.6
C(15)-P(1)-Pd	114.28(10)	C(9)-C(10)-H(10)	118.6
C(2)-P(2)-C(27)	107.76(14)	C(12)-C(11)-C(10)	120.6(3)
C(2)-P(2)-C(33)	105.39(14)	C(12)-C(11)-H(11)	119.7
C(27)-P(2)-C(33)	100.61(13)	C(10)-C(11)-H(11)	119.7
C(2)-P(2)-Pd	113.02(10)	C(13)-C(12)-C(11)	118.4(3)
C(27)-P(2)-Pd	114.57(10)	C(13)-C(12)-H(12)	120.8
C(33)-P(2)-Pd	114.37(10)	C(11)-C(12)-H(12)	120.8
C(43)-N-C(40)	108.0(4)	C(12)-C(13)-C(14)	120.6(3)
C(43)-N-C(39)	117.4(4)	C(12)-C(13)-H(13)	119.7
C(40)-N-C(39)	117.0(4)	C(14)-C(13)-H(13)	119.7
C(43)-N-Pd	68.6(2)	C(13)-C(14)-C(9)	123.2(3)
C(40)-N-Pd	115.9(3)	C(13)-C(14)-H(14)	118.4
C(39)-N-Pd	119.9(3)	C(9)-C(14)-H(14)	118.4
C(3)-B-C(9)	106.5(2)	C(20)-C(15)-C(16)	117.4(3)
C(3)-B-C(1)	109.8(2)	C(20)-C(15)-P(1)	119.1(2)
C(9)-B-C(1)	109.0(2)	C(16)-C(15)-P(1)	123.1(2)
C(3)-B-C(2)	109.1(2)	C(15)-C(16)-C(17)	120.9(3)
C(9)-B-C(2)	109.7(2)	C(15)-C(16)-H(16)	119.5
C(1)-B-C(2)	112.7(2)	C(17)-C(16)-H(16)	119.5
B-C(1)-P(1)	120.3(2)	C(18)-C(17)-C(16)	120.1(3)
B-C(1)-H(1A)	107.3	C(18)-C(17)-H(17)	120.0
P(1)-C(1)-H(1A)	107.3	C(16)-C(17)-H(17)	120.0
B-C(1)-H(1B)	107.3	C(17)-C(18)-C(19)	119.6(3)
P(1)-C(1)-H(1B)	107.3	C(17)-C(18)-H(18)	120.2
H(1A)-C(1)-H(1B)	106.9	C(19)-C(18)-H(18)	120.2
B-C(2)-P(2)	118.7(2)	C(18)-C(19)-C(20)	120.2(3)
B-C(2)-H(2A)	107.6	C(18)-C(19)-H(19)	119.9
P(2)-C(2)-H(2A)	107.6	C(20)-C(19)-H(19)	119.9
B-C(2)-H(2B)	107.6	C(19)-C(20)-C(15)	121.8(3)
P(2)-C(2)-H(2B)	107.6	C(19)-C(20)-H(20)	119.1
H(2A)-C(2)-H(2B)	107.1	C(15)-C(20)-H(20)	119.1
C(8)-C(3)-C(4)	115.3(3)	C(22)-C(21)-C(26)	118.3(3)
C(8)-C(3)-B	125.9(3)	C(22)-C(21)-P(1)	122.8(2)
C(4)-C(3)-B	118.7(3)	C(26)-C(21)-P(1)	118.9(2)
C(5)-C(4)-C(3)	122.4(3)	C(23)-C(22)-C(21)	120.5(3)
C(5)-C(4)-H(4)	118.8	C(23)-C(22)-H(22)	119.7
C(3)-C(4)-H(4)	118.8	C(21)-C(22)-H(22)	119.7
C(4)-C(5)-C(6)	120.4(3)	C(24)-C(23)-C(22)	120.5(3)
C(4)-C(5)-H(5)	119.8	C(24)-C(23)-H(23)	119.7
C(6)-C(5)-H(5)	119.8	C(22)-C(23)-H(23)	119.7
C(7)-C(6)-C(5)	119.2(3)	C(23)-C(24)-C(25)	119.8(3)
C(7)-C(6)-H(6)	120.4	C(23)-C(24)-H(24)	120.1

C(25)-C(24)-H(24)	120.1	C(37)-C(36)-H(36)	120.3
C(26)-C(25)-C(24)	119.8(3)	C(35)-C(36)-H(36)	120.3
C(26)-C(25)-H(25)	120.1	C(36)-C(37)-C(38)	120.5(3)
C(24)-C(25)-H(25)	120.1	C(36)-C(37)-H(37)	119.7
C(25)-C(26)-C(21)	121.0(3)	C(38)-C(37)-H(37)	119.7
C(25)-C(26)-H(26)	119.5	C(37)-C(38)-C(33)	120.7(3)
C(21)-C(26)-H(26)	119.5	C(37)-C(38)-H(38)	119.7
C(32)-C(27)-C(28)	118.5(3)	C(33)-C(38)-H(38)	119.7
C(32)-C(27)-P(2)	121.0(2)	H(39D)-C(39')-H(39E)	109.5
C(28)-C(27)-P(2)	120.5(2)	H(39D)-C(39')-H(39F)	109.5
C(29)-C(28)-C(27)	120.7(3)	H(39E)-C(39')-H(39F)	109.5
C(29)-C(28)-H(28)	119.6	N-C(40)-C(41)	106.5(4)
C(27)-C(28)-H(28)	119.6	N-C(40)-H(40A)	110.4
C(30)-C(29)-C(28)	120.4(3)	C(41)-C(40)-H(40A)	110.4
C(30)-C(29)-H(29)	119.8	N-C(40)-H(40B)	110.4
C(28)-C(29)-H(29)	119.8	C(41)-C(40)-H(40B)	110.4
C(29)-C(30)-C(31)	119.4(3)	H(40A)-C(40)-H(40B)	108.6
C(29)-C(30)-H(30)	120.3	C(40)-C(41)-C(42)	102.5(4)
C(31)-C(30)-H(30)	120.3	C(40)-C(41)-H(41A)	111.3
C(30)-C(31)-C(32)	120.3(3)	C(42)-C(41)-H(41A)	111.3
C(30)-C(31)-H(31)	119.8	C(40)-C(41)-H(41B)	111.3
C(32)-C(31)-H(31)	119.8	C(42)-C(41)-H(41B)	111.3
C(27)-C(32)-C(31)	120.7(3)	H(41A)-C(41)-H(41B)	109.2
C(27)-C(32)-H(32)	119.7	C(43)-C(42)-C(41)	100.0(4)
C(31)-C(32)-H(32)	119.7	C(43)-C(42)-H(42A)	111.8
C(34)-C(33)-C(38)	118.2(3)	C(41)-C(42)-H(42A)	111.8
C(34)-C(33)-P(2)	122.8(2)	C(43)-C(42)-H(42B)	111.8
C(38)-C(33)-P(2)	119.1(2)	C(41)-C(42)-H(42B)	111.8
C(33)-C(34)-C(35)	120.7(3)	H(42A)-C(42)-H(42B)	109.5
C(33)-C(34)-H(34)	119.6	N-C(43)-C(42)	111.5(4)
C(35)-C(34)-H(34)	119.6	N-C(43)-Pd	73.1(2)
C(36)-C(35)-C(34)	120.5(3)	C(42)-C(43)-Pd	116.8(3)
C(36)-C(35)-H(35)	119.7	N-C(43)-H(43)	116.1
C(34)-C(35)-H(35)	119.7	C(42)-C(43)-H(43)	116.1
C(37)-C(36)-C(35)	119.3(3)	Pd-C(43)-H(43)	116.1

Table 19. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for 9 (CCDC 186800). The anisotropic displacement factorexponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd	266(1)	269(1)	153(1)	-45(1)	44(1)	47(1)
P(1)	236(5)	174(4)	163(4)	-26(3)	64(3)	18(3)
P(2)	221(4)	190(4)	151(4)	-17(3)	85(3)	6(3)
N	320(20)	850(30)	490(20)	170(20)	-92(18)	-80(20)
C(43')	320(20)	850(30)	490(20)	170(20)	-92(18)	-80(20)
B	218(18)	200(19)	161(16)	-20(15)	69(14)	-6(16)
C(1)	240(16)	198(16)	181(15)	6(14)	103(13)	55(15)
C(2)	222(16)	155(16)	193(15)	-1(13)	87(13)	7(14)
C(3)	218(17)	189(17)	193(16)	10(13)	61(13)	19(14)
C(4)	380(20)	201(18)	177(16)	18(13)	67(15)	12(15)
C(5)	520(20)	320(20)	181(17)	-59(15)	56(17)	31(18)
C(6)	430(20)	250(20)	320(20)	-101(16)	14(18)	-64(17)
C(7)	287(19)	269(19)	400(20)	6(17)	77(16)	-54(17)
C(8)	250(18)	274(19)	245(17)	-36(15)	82(15)	-6(15)
C(9)	227(17)	218(17)	125(15)	-62(12)	91(13)	4(14)
C(10)	243(19)	284(19)	231(17)	-39(14)	92(15)	31(14)
C(11)	275(19)	350(20)	220(17)	-48(15)	72(15)	105(16)
C(12)	490(20)	250(19)	174(17)	32(14)	158(16)	149(17)
C(13)	430(20)	237(19)	248(18)	14(14)	214(17)	27(16)
C(14)	265(18)	250(18)	191(16)	-20(13)	126(14)	36(14)
C(15)	214(17)	180(17)	199(16)	2(13)	65(13)	20(14)
C(16)	330(20)	260(20)	360(20)	-90(16)	133(17)	-14(16)
C(17)	350(20)	360(20)	450(20)	-94(18)	221(19)	37(18)
C(18)	214(19)	350(20)	370(20)	60(17)	119(16)	62(16)
C(19)	254(18)	290(20)	348(19)	3(16)	48(15)	-42(16)
C(20)	300(18)	228(17)	271(17)	-65(16)	81(14)	4(17)
C(21)	258(18)	204(17)	226(17)	13(13)	114(14)	0(14)
C(22)	265(19)	236(18)	245(18)	-6(14)	81(15)	30(15)
C(23)	360(20)	270(20)	288(19)	-52(15)	94(16)	-24(16)
C(24)	540(30)	290(20)	410(20)	-44(17)	190(20)	-172(18)
C(25)	800(30)	350(20)	290(20)	59(17)	230(20)	-140(20)
C(26)	620(30)	310(20)	211(18)	-39(15)	156(18)	-130(19)
C(27)	239(17)	179(16)	153(15)	-29(12)	109(13)	-14(13)
C(28)	235(18)	230(18)	188(16)	-43(13)	112(14)	-22(14)
C(29)	275(19)	246(19)	216(17)	-5(14)	102(15)	63(15)
C(30)	390(20)	209(18)	288(19)	25(14)	172(17)	-8(16)
C(31)	280(20)	300(20)	380(20)	21(16)	158(17)	-51(16)
C(32)	234(18)	277(19)	264(18)	54(14)	112(15)	43(15)
C(33)	241(17)	190(17)	178(15)	23(13)	119(13)	56(14)
C(34)	261(18)	260(20)	175(15)	2(13)	78(14)	12(14)
C(35)	276(19)	360(20)	274(18)	77(15)	143(16)	27(15)
C(36)	370(20)	370(20)	219(17)	91(15)	183(16)	137(17)
C(37)	470(20)	330(20)	207(18)	-67(15)	175(17)	7(18)
C(38)	360(20)	300(20)	279(19)	-60(15)	172(17)	-38(16)
C(39)	520(30)	290(30)	450(30)	140(20)	-120(20)	140(20)

C(40)	770(40)	900(40)	660(40)	230(30)	170(30)	-40(30)
C(41)	780(40)	1080(50)	390(30)	0(30)	90(30)	-620(30)
C(42)	890(40)	790(40)	390(30)	-200(20)	310(30)	-380(30)
C(43)	780(30)	650(30)	300(20)	-160(20)	120(20)	-120(30)
N'	780(30)	650(30)	300(20)	-160(20)	120(20)	-120(30)

Table 20. Hydrogen coordinates (x 10^3) and isotropic displacement parameters ($Å^2$x 10^3) for 9 (CCDC 186800).

	x	y	z	U_{iso}
H(43')[b]	-206	171	-33	76
H(1A)	91	143	578	24
H(1B)	60	99	492	24
H(2A)	354	113	413	23
H(2B)	271	158	369	23
H(4)	278	150	766	32
H(5)	377	209	899	44
H(6)	493	263	841	45
H(7)	508	256	647	40
H(8)	402	198	508	31
H(10)	485	93	657	30
H(11)	571	27	767	35
H(12)	442	-30	792	36
H(13)	225	-22	693	34
H(14)	139	43	577	27
H(16)	-119	220	509	38
H(17)	-289	211	567	44
H(18)	-431	152	484	38
H(19)	-399	99	348	38
H(20)	-229	107	294	33
H(22)	124	222	584	31
H(23)	186	299	605	38
H(24)	136	345	428	49
H(25)	21	315	226	57
H(26)	-34	238	203	46
H(28)	351	27	374	25
H(29)	352	-49	427	29
H(30)	164	-88	385	34
H(31)	-26	-49	288	37
H(32)	-28	29	240	31
H(34)	368	142	222	28
H(35)	458	136	74	35
H(36)	389	81	-84	36
H(37)	223	33	-100	39
H(38)	125	41	40	36
H(39A)[a]	-130	197	-41	62
H(39B)[a]	-259	206	-26	62
H(39C)[a]	-259	179	-148	62
H(39D)[b]	-152	169	-102	38
H(39E)[b]	-207	126	-194	38
H(39F)[b]	-60	136	-136	38
H(40A)	-376	123	-81	98
H(40B)	-334	130	68	98
H(41A)	-351	50	-19	96
H(41B)	-239	62	115	96
H(42A)	-118	39	8	80

H(42B)	-221	56	-126	80
H(43) [a]	-78	119	-67	74

[a] Population = 0.815(7)
[b] Population = 0.185(7)